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ABSTRACT

This technical specialty course is one of thirty-five courses designed for use by two-year postsecondary institutions in five nuclear technician curriculum areas: (1) radiation protection technician, (2) nuclear instrumentation and control technician, (3) nuclear materials processing technician, (4) nuclear quality-assurance/quality-control technician, and (5) nuclear reactor (plant) operator trainee. This course, Radiation Monitoring Techniques (Radiochemical), contains seven modules: Radiation Sample Preparation, Liquid; Radiation Sample Preparation, Solid; Air Monitoring Techniques, Particulate; Air Monitoring Techniques, Gaseous; Water Monitoring Techniques, Dissolved Solids; Water Monitoring Techniques, Suspended Solids; and Radiation Monitoring of Biological Samples. Each module follows a typical format including the following sections: introduction, module prerequisites, objectives, subject matter, exercises, materials, procedures, data tables, reference materials, and tests. (A master index and suggested program planning guides are also available--see CE 026 856 and CE 026 639-643.) (LRA)

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Nuclear Technology Series

COURSE 20

RADIATION MONITORING TECHNIQUES (RADIOCHEMICAL)

CENTER FOR OCCUPATIONAL RESEARCH

AND DEVELOPMENT, INC.

4800 LAKEWOOD DRIVE

WACO, TEXAS 76710

CE 026 660

U.S. DEPARTMENT OF HEALTH,
EDUCATION & WELFARE
NATIONAL INSTITUTE OF
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PREFACE

ABOUT NUCLEAR TECHNOLOGY MODULES

The modules – developed for use in two-year postsecondary technical institutions to prepare technicians for employment – are useful in industry for updating employees in company-sponsored training, and as laboratory texts in engineering schools. The principles, techniques and skills taught in the modules, based on tasks that nuclear technicians perform, were obtained from a nationwide advisory committee of employers of nuclear technicians. Each module was written by a working technical expert/experienced writer team and tested with students.

A module contains the following elements:

Introduction, which identifies the topic and often includes a rationale for studying the material.

Module Prerequisites, which identify the material a student should be familiar with before studying the module.

Objectives, which clearly identify what the student is expected to know for satisfactory module completion. The objectives, stated in terms of action-oriented behaviors, include such action words as operate, measure, calculate, identify and define, rather than words with many interpretations, such as know, understand, learn and appreciate.

Subject Matter, which presents the background theory and techniques supportive to the objectives of the module. Subject matter is written with the technical student in mind.

Exercises, which provide practical problems to which the student can apply this new knowledge.

Materials, which identify the equipment required to complete the laboratory procedure.

Procedures, which is the experiment section, or "hands-on" portion of the module (including step-by-step instruction) designed to reinforce student learning.

Data Tables, which are included in most modules for the first year (or basic) courses to help the student learn how to collect and organize data.

Reference Materials, which are included as suggestions for supplementary reading/viewing for the student.

Test, which measures the student's achievement of the prestated objectives.

A helpful Index has been added at the end of each course.

The materials contained herein were developed pursuant to a contract with the Office of Education, U.S. Department of Health, Education and Welfare: Contract No. 300780582. Contractors undertaking such projects under Government sponsorship are encouraged to express freely their professional judgment in the conduct of the project. Points of view or opinions stated do not, therefore, necessarily represent official Office of Education position or policy.

The Nuclear Technology Series consists of five curricula:

- Nuclear Reactor (Plant) Operator Trainee.
- Radiation Protection Technician.
- Nuclear Materials Processing Technician.
- Nuclear Quality-Assurance/Quality-Control Technician.
- Nuclear Instrumentation and Control Technician.

The completed Nuclear Technology Series consists of the following 35 technical specialty courses:

- Radiation Physics.
- Radiation Protection I.
- Principles of Process Instrumentation.
- Heat Transfer and Fluid Flow.
- Introduction to Nuclear Systems.
- Instrumentation and Control of Reactors and Plant Systems.
- Reactor Operations.
- Reactor Safety.
- Reactor Auxiliary Systems.
- Power Plant Systems.
- Radiation Detection and Measurement.
- Reactor Physics.
- Power Plant Chemistry.
- Introduction to Quality Assurance/Quality Control.
- Metallurgy and Metals Properties.
- Mechanical Component Characteristics and Specifications.
- Radiation Protection II.
- Radiological Emergencies.
- Radiation Shielding.
- Radiation Monitoring Techniques (Radiochemical).
- Radioactive Materials Disposal and Management.
- Advanced Radionuclide Analysis.
- Nuclear Chemical Processes.
- Nuclear Systems and Safety.
- Radioactive Material Handling Techniques.
- Nondestructive Examination (NDE) Techniques I.
- Metrology.
- Welding Inspection.
- Civil/Structural Inspection.
- Mechanical Inspection.
- Quality-Assurance Practices.
- Nondestructive Examination (NDE) Techniques II.
- Control Systems I.
- Control Systems II.
- Power Plant Electrical Systems and Components.

Availability: Instructional modules are available individually or by courses from:

Center for Occupational Research and Development
4800 Lakewood Drive, Suite 5
Waco, Texas 76710
Telephone (817) 772-8756

Printed:

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MODULE 20-7 Radiation Monitoring of Biological Samples

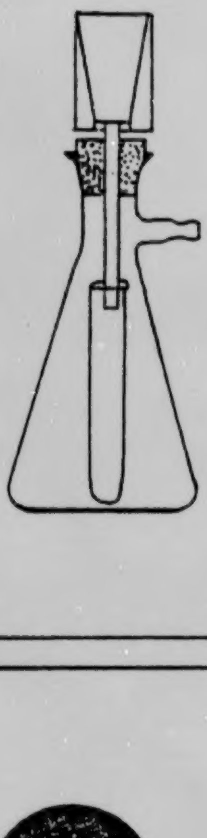
NUCLEAR TECHNOLOGY

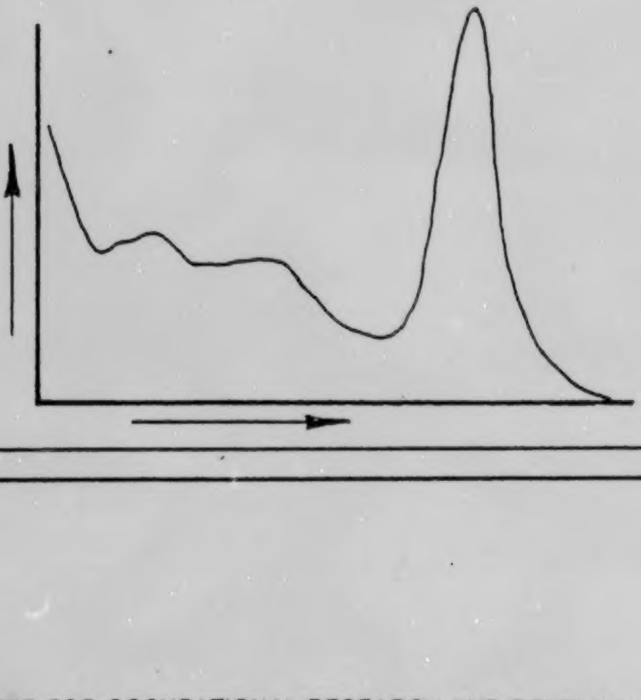
MODULE 20 - 1

RADIATION SAMPLE PREPARATION - LIQUID

RADIATION MONITORING
TECHNIQUES (RADIOCHEMICAL)

COURSE 20





CORD CENTER FOR OCCUPATIONAL RESEARCH AND DEVELOPMENT

COURSE MODULES

COURSE 20	RADIATION MONITORING TECHNIQUES (RADIOCHEMICAL)
⇨ MODULE 20-1	Radiation Sample Preparation - Liquid
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MODULE 20-7	Radiation Monitoring of Biological Samples

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INTRODUCTION

Safe and efficient operation of a radiochemical laboratory depends entirely upon the personnel. Any person who works in a radiochemical laboratory must be willing to abide by the rules of operation to ensure safe activities in the laboratory. Anyone who is careless in procedures will endanger the entire laboratory and staff.

Proper techniques with appropriate precautions will allow workers to use any reasonable amount of radioactive materials without danger to themselves, their co-workers, or the facility.

This module explains rules that apply to both the general laboratory and the radiation laboratory. Also discussed are special apparatus and equipment; weighing and measurement of liquid volumes; remote handling; and sample mounts, drying, and concentration.

MODULE PREREQUISITES

The student should have completed Course 1, "Radiation Physics"; and Course 11, "Radiation Detection and Measurement."

OBJECTIVES

Upon completion of this module, the student should be able to:

1. Identify and explain the use of standard laboratory apparatus.
2. List general rules that would apply to any laboratory in which chemicals are used.
3. List specific safety precautions to be used when working with radioactive materials in the laboratory.
4. List procedures required for routine care and maintenance of radiochemical laboratory equipment.
5. Explain procedure for using disposable laboratory ware.
6. Use appropriate equipment and micromeasurement techniques to prepare a liquid for counting.

SUBJECT MATTER

LABORATORY SAFETY

Radiochemical laboratories are much safer than the average home. This statement is true because radiochemical laboratories and their contents have been designed for safe operation, and the scientists and technicians employed in them have been trained to consider safety as an important aspect of their daily routine. Although safe practices become almost routine to the well-trained radiochemical worker, there must be continued re-enforcement to ensure a lasting conscious application in all aspects of operation.

Good laboratory safety practices are just as important to an employer as to an employee. To an employer, an accident means lost time, equipment, materials, and possibly employees. Because of this, laboratories are provided with equipment designed to prevent accidents and to prevent or reduce injury or damage that would result should an accident occur.

GENERAL LABORATORY RULES

Each laboratory should have a set of rules posted, which, if followed, will reduce probability or seriousness of an accident that might occur. Each employer is required to inform each employee of these rules, but the employee is obligated to herself or himself and to co-workers to learn them and to apply them.

DO NOT WORK ALONE IN ANY LABORATORY

This is the first rule of safety for any laboratory. To work alone in a lab is to invite disaster, should an accident occur. Any person working alone might not hear or pay attention to alarms. Additionally, potential rescuers may not know the location, or even the presence, of a lone, incapacitated worker.

The second general rule is –

KNOW HOW TO REPORT A FIRE AND KNOW THE LOCATION OF FIREFIGHTING EQUIPMENT

Naturally, when a fire is first detected, it should be extinguished with the equipment at hand if possible. However, if this is not possible, the employee should report the fire and sound the alarm to evacuate the building.

USE SAFETY EQUIPMENT

Safety equipment is necessary for protection of both the individual and property. Use of such equipment is required by and enforced by the Occupational Safety and Health Act (OSHA).

LABEL ALL MATERIALS

Never use any material not properly identified by a label. Any material not labeled must be considered to be unidentified and extremely hazardous.

REPORT ALL ACCIDENTS

All accidents, no matter how trivial, must be reported for two reasons. First, the report may help to prevent reoccurrence of the same kind of accident. Second, should further complications develop after the accident, the employee will be on record as having reported the accident. Thus, by reporting all accidents, the employee helps himself or herself, other employees, and the employer.

These rules can be applied to any situation and must be applied scrupulously by all who work in the nuclear industry.

SPECIAL SAFETY RULES

There are some special safety rules from chemical and electronics laboratories that also apply to radiation laboratories.

CHEMICAL RULES

In addition to the usual rules of a chemistry laboratory, the following rules must be strictly enforced in a radiation laboratory.

- Never pipette by mouth suction.
- Use double barriers to ensure containment.
- Keep containers closed when not in use.
- Keep fume hood running when it is in use.
- Use catch pans and absorbent paper when handling liquids.
- Avoid skin contact.
- Report all spills immediately.

PHYSICAL RULES

Rules concerning physical aspects of the laboratory are especially applicable to radiation laboratories. Although these rules consist generally of good housekeeping, they cannot be stressed too often.

- Keep the work area uncluttered.
- Keep electrical cords in good repair.
- Make electrical connections only with the equipment turned off.
- Identify high-voltage hazards.

SPECIFIC RADIATION LABORATORY RULES

All laboratories have some rules that, more or less, are rigidly enforced. Laxity cannot be permitted in a radiation laboratory. All rules must be applied at all times.

The previously mentioned rules are for the health and safety of those who work in the laboratory. The rules that follow are not only for health and safety of the worker, but also for those with whom the laboratory workers may live.

- Always wear personal monitoring equipment when in the laboratory.
- Wear suitable protective clothing.
- Do not eat, store, or prepare food in the laboratory.
- Do not smoke in the laboratory.
- Place liquid radioactive waste in appropriate labeled containers.
- Place solid radioactive waste in the trash can labeled "radioactive waste."
- Remove protective gloves and coats before entering the counting room.
- Monitor hands, shoes, and clothing when leaving the radiation laboratory.
- Listen to and believe the monitors.
- Any person who knowingly swallows, inhales, or receives an injection of radioactive material or who has been overexposed must report to the supervisor and then to a competent physician immediately.
- All spills must be cleaned and decontaminated immediately to permissible levels.
- Record all receipt, transfer, and disposal of radioactive materials.
- Store all radioactive materials in appropriate containers in their designated areas.
- When in doubt about a procedure or material, ask the radiation safety officer.

Strict adherence to these rules will help to ensure a safe working environment for workers in a radiation laboratory.

SPECIAL APPARATUS AND EQUIPMENT

A complete listing and discussion of all special apparatus and equipment used in a radiochemical laboratory is not possible in a text of this nature. However, a few of the more important items will be considered. Inclusion in this discussion does not mean that these items are considered the most important.

RADIOISOTOPE HOODS

Radioisotope hoods are somewhat different from the usual chemical fume hoods. The hood should be lined with stainless steel for ease of decontamination. The work surface should be reinforced to support lead shielding, as it may be needed. Such units may be required to support as much as 200 pounds per square foot for shielding.

The exhaust system should be equipped with a high-efficiency filter to remove particulates in the 0.3- to 0.5-micrometer range. The blower for the exhaust system should provide for face-air velocities of 46 to 61 meters per minute (150 to 200 ft/min). Face-air velocities are measured at the entrance to the fume hood.

The radioisotope fume hood should be at least 1.3 meters wide, but preferably 3 meters. Figure 20-1-1 illustrates a typical radioisotope hood.

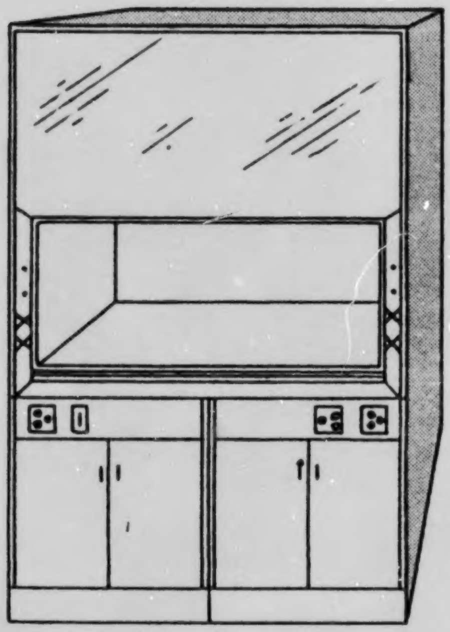


Figure 20-1-1. A radioisotope hood.

MICROLITER PIPETTES

Volumes of liquids used in working with radioactive materials may be very small. The unit used to describe these small volumes is the microliter (μl), the term for one-millionth of a liter. Another term often employed for the same volume is lambda (λ).

Dispensing of such a small volume requires a pipette with a very thin bore. This pipette is calibrated to contain (not to deliver) the indicated volume. Therefore, it must be rinsed, and the rinsings must be added to the measured sample.

These small volumes are manipulated by use of a micropipette control. The micropipette control may be a microliter syringe, a tuberculin syringe, or a device that regulates volume of liquid in the pipette by means of a screw-type collar called a "Clay Adams pipetter." Any of these three can be manipulated easily with one hand. Figure 20-1-2 illustrates a micropipette (a) and Clay Adams control (b).

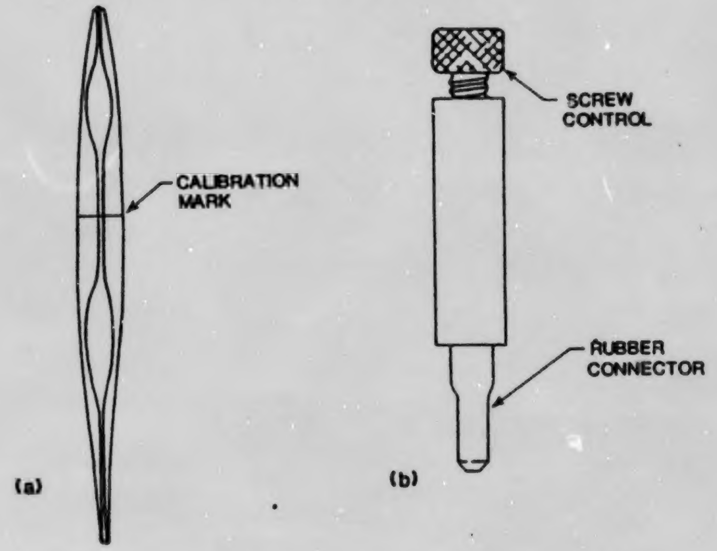


Figure 20-1-2. Micropipette (a) and Clay Adams control (b).

During the past several years, many micropipetting systems have been developed. These systems consist of piston-type pipettes to which disposable, non-wettable plastic tips are attached. These units are manufactured to pipette a single volume and are available in volumes of 5, 10, 15, 20, 25, 30, 40, 50, 60, 70, 75, 80, 90, 100, 120, 150, 200, 250, 300, 400, 500, 600, 700, 750, and 1000 µl. The volume pipetted is determined by cylinder diameter and piston-stroke length. This volume is fixed for each pipette, and it cannot be altered. Total stroke of the piston is divided into two stages. The first stage determines working volume, and the second stage provides for blowout of liquid remaining in the tip (Figure 20-1-3).

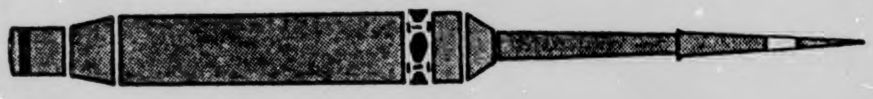


Figure 20-1-3. Eppendorf pipette and tips.

REMOTE PIPETTES

Often, samples are so radioactive that small quantities must be transferred remotely. These occasions will necessitate use of a remote pipetting device such as that depicted in Figure 20-1-4. These devices normally are operated over the top of, or through a port in, a shield of some type, although distance may serve as sufficient shielding. Use of these tools requires skills that can be developed only by practice. They often must be used in conjunction with remote or reflected viewing.

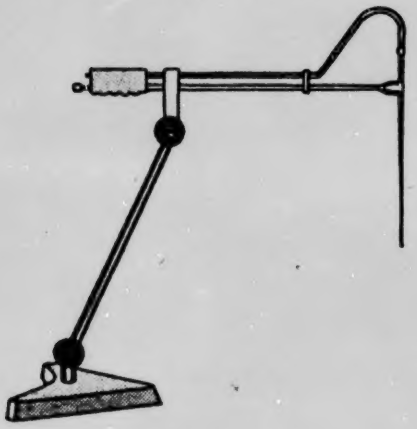


Figure 20-1-4. Remote pipetter and stand.

The remote pipette device usually consists of a carrier arm equipped with a finger, to which the pipette is attached. The arm is equipped with appropriate handles, a pipette control (which may be a syringe or some other plunger-actuated device), and a tube with which the pipette is connected to the pipette control.

Actual operation of these units usually is limited to transfer of approximate volumes, and cannot be construed as precision measurement. However, in many cases, approximation may be sufficient. At the very least, use of a smaller amount of material may reduce the radiation level to a point at which a precision micropipette or regular pipette can be employed with standard techniques.

REMOTE-HANDLING DEVICES

Remote-handling devices include everything from small forceps to giant robot hands. Typically, a lab will be equipped with a series of tongs and tools for the picking up and handling of objects from distances that range from 1 foot to 5 feet. These devices may be equipped with a variety of jaws for either general or specific uses. Figure 20-1-5 illustrates several types of remote-handling devices.

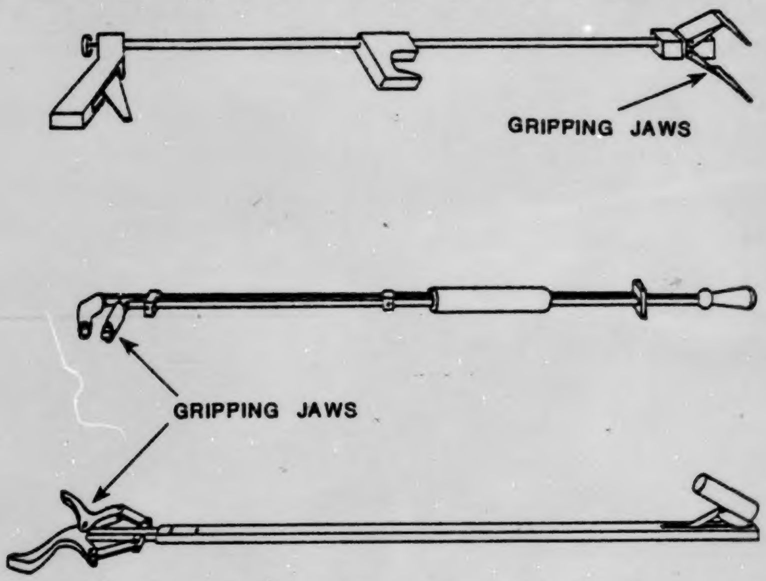


Figure 20-1-5. Remote-handling tongs.

DISPOSABLE LABWARE

Disposable labware is certainly a boon in the radiation chemistry laboratory. Such labware reduces radiation exposure levels and the many hours required for decontamination of labware. However, there are certain precautions to be observed in the use of disposable materials.

Material From Which Constructed

First consideration should be for the material from which the disposable item is constructed. The prime concern should be whether or not the material will be harmed by either the temperature of the material or solvents that may be present.

Calibration and Accuracy

The second consideration should be for calibration and accuracy. Many disposable items are mass produced and should be checked routinely for calibration and accuracy. This does not mean that every item must be checked, but that each lot of items should be checked if calibration and accuracy are important. Most will be found adequate, but a lot must be rejected if it is found to be outside of acceptable calibration limits.

Disposal

Disposable labware, contaminated with radioactive materials, requires special attention. The simplest procedure is to dispose of the item by placing it in the waste receptacle labeled "radioactive waste." Although this is acceptable, some laboratories require additional steps. Occasionally, a laboratory will require that disposable items be monitored for activity levels before disposal. Such monitoring requires a log in which to record the item, approximate count rate, and the isotope of contamination. Even more rare is the requirement that glass items be broken during disposal. When breakage is required, an item on which the glass can be broken is provided in the waste receptacle. The object of breakage is the prevention of any possible re-use of the glassware. However, this is usually not a problem.

Unless the contamination levels are excessively high, disposable labware simply is placed in the radioactive waste container. When contamination levels are high, Health Physics should be notified, either to determine procedures or for special packaging and handling in order that the department can determine any special procedures or can decide upon special packaging and handling.

WEIGHING

Samples to be weighed in the radiochemical laboratory usually range from a few grams down to milligram quantities, even smaller in some cases. Balances normally found in a radiochemistry laboratory are of two types, top-loading balances and analytical balances.

TOP-LOADING BALANCES

Top-loading balances have proved to be very useful in many laboratories. They are reliable, convenient, easy to operate, and require little or no special training of the user. Accessibility of the pan, coupled with a large tare range, makes routine weighing of samples very easy. Figure 20-1-6 illustrates a top-loading balance and its controls.

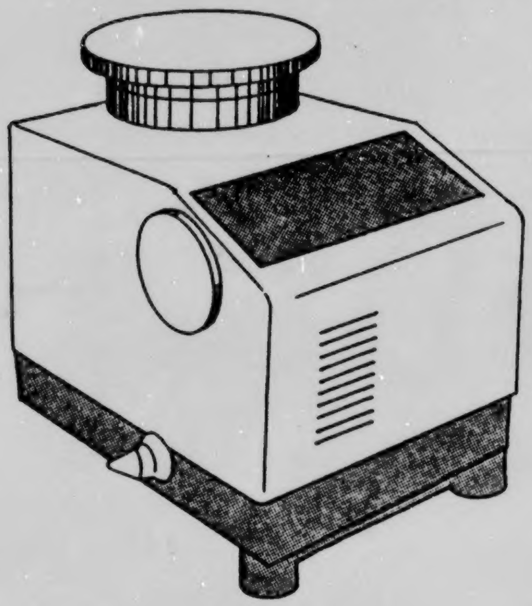


Figure 20-1-6. Top loading balance.

ANALYTICAL BALANCES

The modern analytical balance is still essentially a first-class lever, damped in some manner to reduce oscillations, for comparison of an unknown mass with known masses. The known masses are contained within the case and are placed for comparison by use of external controls. The unknown is placed on the pan of the balance, and weights or comparison masses simply are dialed in. Figure 20-1-7 illustrates an analytical balance.

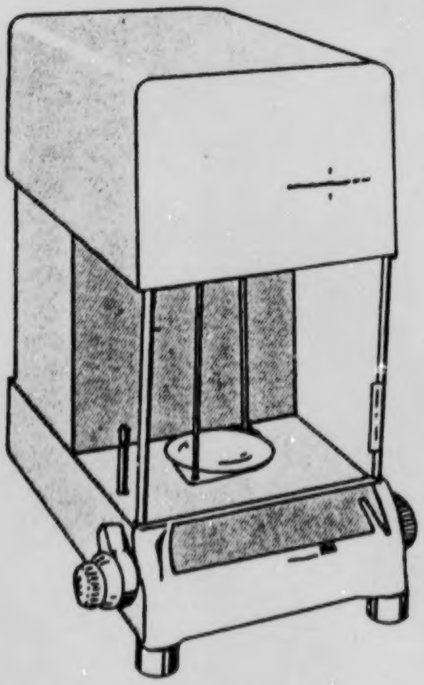


Figure 20-1-7.
Analytical balance.

Several rules must be observed in the use of any analytical balance. Strict adherence to these rules will ensure many years of use.

- Keep the environment of the balance clean and neat.
- Handle instrument with care.
- Keep balance case closed when instrument is not in use.
- Ensure that balance is arrested before adding or removing weights or material.
- Hot objects, moist objects, and liquids must not be allowed to come in contact with the pan.

- Objects must be at the temperature of the balance when they are weighed.
- Close balance door before making final reading.

MEASURING LIQUID VOLUMES

Skill in volumetric measurement is essential in radiochemistry because these measurements must be both precise and accurate. Preparation of solutions of known activity concentration is necessary for many applications. Likewise, solutions of unknown activity may need to be diluted to a known volume for identification and concentration determination.

Preparation of a solution of known activity concentration is a reasonably simple operation. Assuming that the radioactive material originally is received in solution and that the activity per milliliter is known, a volume to produce required total activity would be removed from the sample, placed in an appropriate volumetric flask, and diluted to volume.

Procedure for preparation of a solution of known activity concentration should be step-wise as follows:

1. Calculate total activity required in final solution according to Equation 20-1-1.

$$\begin{array}{c}\text{total}\\\text{activity}\end{array} = \begin{array}{c}\text{activity/}\\\text{unit volume}\end{array} \times \begin{array}{c}\text{total}\\\text{volume}\end{array}$$

Equation 20-1-1
(Equation = Eq.)

2. Calculate volume of original solution that contains required total acitivty according to Equation 20-1-2.

$$\text{required volume} = \text{total activity} \times \frac{\text{volume}}{\text{original activity}} \qquad \text{Eq. 20-1-2}$$

3. Fill a volumetric flask of appropriate size approximately 2/3 full with diluting solution.
4. Quantitatively transfer required volume (calculated by Equation 20-1-2) to liquid in the volumetric flask.
5. Finish filling volumetric flask to the mark with appropriate diluting solution.
6. Mix solution in capped volumetric flask by inverting and righting at least 10 times.
7. Transfer solution to a clean, stoppered container, or leave it in the volumetric flask as required; label container with identity of the solution, activity of the solution, the isotope, date, and name of person who prepared solution.

EXAMPLE

Given: One milliliter of a solution containing 900 microcuries of ^{14}C.

Find: (a) Required total activity in the final dilution and (b) volume of the original solution that contains the required total activity to prepare 25 milliliters of solution containing 0.9 microcurie per milliliter.

Solution: (a) Use Equation 20-1-1:

$$\text{total activity} = \text{activity/milliliter} \times \text{number of milliliters}$$

$$= 0.9\ \mu\text{Ci/ml} \times 25\ \text{ml}$$

$$= 22.5\ \mu\text{Ci}.$$

(b) Use Equation 20-1-2:

$$\text{required volume} = \text{total activity} \times \frac{\text{volume}}{\text{original activity}}$$

$$= 2.25\ \mu\text{Ci} \times \frac{1\ \text{ml}}{900\ \mu\text{Ci}}$$

$$= 0.025\ \text{ml}$$

$$= 25\lambda.$$

Preparation of solutions of known dilution follows a somewhat similar procedure. Steps involved are as follows:

1. Determine amount of dilution required; this is a decision required of the worker (whether to dilute the original solution 10 times, 100 times, 1000 times, or more, or in between).
2. Select an appropriate volumetric flask to produce final volume required.
3. Calculate volume of the original solution required to produce dilution in the volumetric flask according to Equation 20-1-3.

$$\text{volume required} = \text{flask volume} \times \text{dilution factor} \qquad \text{Eq. 20-1-3}$$

4. Fill volumetric flask approximately 2/3 full of diluting solution.
5. Quantitatively transfer required volume of original solution (as calculated from Equation 20-1-3) to solution in the flask.
6. Finish filling volumetric flask to the mark with diluting solution.
7. Mix solution thoroughly by inverting and righting the capped volumetric flask at least 10 times.
8. Transfer solution to a clean, stoppered flask, or leave it in volumetric flask as required; label container with identity of solution, the isotope, the date, and name of person who prepared the solution.

EXAMPLE

Given: 50 milliliters of a 1:10,000 dilution of a solution containing 30 microcuries of ^{137}Cs/ml.

Find: Volume of original solution required.

Solution: Use Equation 20-1-3:

$$\begin{aligned}\begin{matrix}\text{volume}\\\text{required}\end{matrix} &= \begin{matrix}\text{flask}\\\text{volume}\end{matrix} \times \begin{matrix}\text{dilution}\\\text{factor}\end{matrix}\\ &= 50\ \text{ml} \times \frac{1}{10{,}000}\\ &= 0.005\ \text{ml}\\ &= 5\lambda.\end{aligned}$$

REMOTE HANDLING

Remote handling of large activities of beta/gamma emitters can become a very specialized and complicated subject. Use of tongs of various kinds in the radiation laboratory is essential and may have to be combined with shielding if distance does not reduce dose rate sufficiently. Always consider that no source should be handled with the fingers. Remember this: Calculated dose from 1μCi of ^{32}P (1/10th the license-exempt quantity) is 3 R per hour at 3 millimeters. Use of tweezers or tongs will reduce exposure significantly.

Consider what happens when a shipment of 10 mCi of 1-MeV gamma emitter is unpacked. Assuming the top of the pig to be 2.5 cm from the source, the dose to hands at the opening of the pig would be approximately 900 mr per hour. Use of 30-cm tongs will reduce dose to approximately 6 mr per hour.

Use of these handling devices may be complicated by having to work over or through a barrier wall. Observation may be through lead-glass, by mirror, or by closed-circuit television. Skill in these manipulations is obtained best by practice with nonradioactive materials. Some of the activities in the Procedures section of this module will involve both remote handling and remote viewing.

SAMPLE MOUNTS

Radioactive samples usually are placed in some type of holder or mount before they are counted. There are three types of mounts in general use. These are card mounts, planchets, and ring-and-disc sets.

CARD MOUNTS

The simplest and least expensive of these mounts is the card mount. This mount usually is reserved for an infinitely thin sample or a relatively thin sample on filter paper.

This type of mount may be constructed of a rectangle of cardboard, plastic, or metal, 1/2 to 1 millimeter in thickness, with a hole, 25 millimeters in diameter, bored in its center. A thin plastic film is placed over the hole and taped firmly into position. The thin plastic film typically has a thickness of 2 mg/cm^2 or less. When the mount is in its upright position, the plastic film is on the bottom, and the hole resembles a shallow well. The sample is placed on the plastic film. A second plastic film may be placed over the sample and taped to the card, thus sealing the sample between the two films. Figure 20-1-8 is an illustration of a card mount.

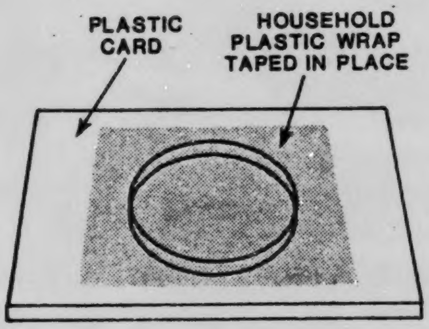


Figure 20-1-8.
Card mount.

PLANCHET MOUNTS

Planchets are small, shallow cups that contain samples. They are available in a variety of sizes, shapes, and materials. Common materials include glass, plastic, aluminum, and stainless steel. Liquid samples either can be placed in planchets and dried or counted as a liquid. Filter papers can be placed in planchets, and solid samples can be placed directly in the planchets. Figure 20-1-9 illustrates several types of planchets.

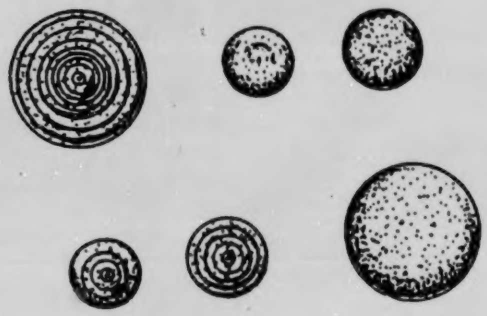


Figure 20-1-9.
Planchets.

RING-AND-DISC SETS

Occasionally, to electroplate or otherwise deposit a sample on a piece of metal for counting may be desirable. Such processes may be desirable as a means of separation, for backscatter characteristics, or as a means of collection. For whatever reason, suitable discs are available in a variety of metals.

The advantage of a sample that has been electroplated on a disc is that the sample then can be counted without a protective cover, with little fear of the radioactive material being wiped off.

A ring-and-disc set is prepared as follows: The active material is deposited on the disc; the disc is placed on a carrier backing; and the ring is snapped onto the carrier, over the disc. Thus, the disc is held in place by the ring. Figure 20-1-10 illustrates a carrier disc and ring.

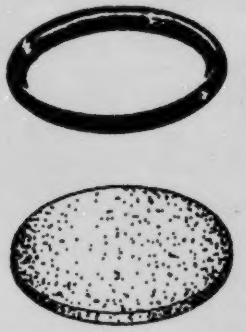


Figure 20-1-10.
Ring-and-disc set.

SAMPLE DRYING

Liquid samples placed in planchets often must be dried before they are counted. Drying can be accomplished efficiently with an infrared lamp held by a burette clamp on a ringstand over the sample. A more even and reproducible distribution of the sample can be obtained by use of a rotating platform below the heat lamp. With this apparatus the samples are turned continuously while drying. (Refer to Figure 20-1-11.)

There is a great temptation to hasten the drying process by placing the lamp too close to the sample. This practice can cause warping of plastic planchets, splatter and loss of sample by boiling, or even complete evaporation of the sample.

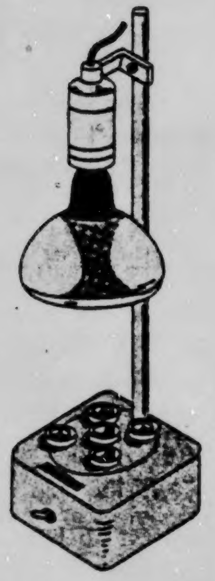


Figure 20-1-11. Heat lamp and sample spinner.

LIQUID-SAMPLE CONCENTRATION

Low levels of radioactivity in solution can be determined by evaporation of the solution in order to concentrate the radionuclides from a known volume. A liquid-concentration unit is depicted in Figure 20-1-12. This unit is designed to evaporate liquid at a controlled rate and to retain and concentrate soluble salts on the surface of a 4-cm aluminum planchet. The unit consists of an aluminum evaporating dish with a sloping inside surface, an aluminum cover, and an electric heating element controlled by a variac. An aluminum plug having a recess in one face to accommodate a flat aluminum planchet is located centrally in the bottom of the dish.

This technique requires use of a silicone grease on all evaporation surfaces to localize residue in an area 2.5 cm in diameter in the center of the planchet. When the liquid has

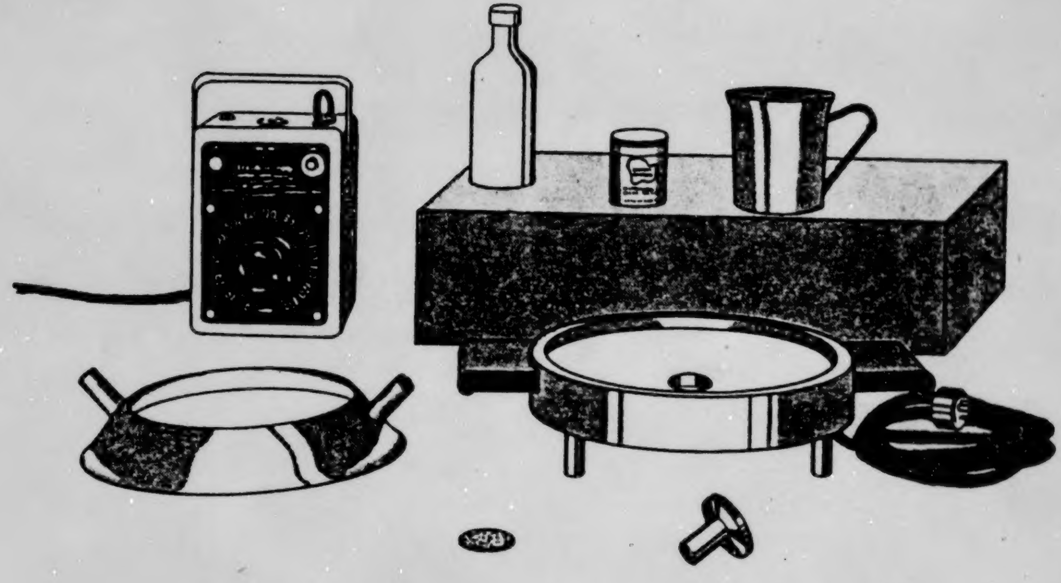


Figure 20-1-12. Liquid concentration unit.

evaporated completely, the planchet is removed from the apparatus and counted. This unit will evaporate approximately 1 liter of water in 3 1/2 hours.

The technician can determine activity per unit volume of the liquid by counting the sample, deriving its activity, and dividing the activity by total volume of the liquid. This calculation is accomplished with Equation 20-1-4.

$$\text{activity } \mu\text{Ci/ml} = \frac{\text{total activity in } \mu\text{Ci}}{\text{volume of sample in ml}} \qquad \text{Eq. 20-1-4}$$

NONDESTRUCTIVE TECHNIQUES

The assay techniques described in this module are of the destructive type. Some of the modern analytical techniques involve nondestructive assay instruments such as fast and thermal neutron counters, segmental gamma scanners, calorimeters, and solution assay units.

EXERCISES

1. Write the procedure for use of the analytical balance available.
2. Write the procedure for preparation of a 10-ml solution containing 1.5 µCi/ml from an original solution containing 30 µCi/ml.
3. Write the procedure for preparation of a 50-ml solution containing 0.5 µCi/ml from an original solution containing 700 µCi/ml.
4. Write the procedure for preparation of a 1:5000 dilution of a solution that contains 2.5 mCi of ^{51}Cr per ml.
5. With Equation 20-1-4, calculate activity in µCi/ml of a sample of water found to contain 7 µCi of ^{60}Co in 500 ml of liquid.

MATERIALS

Lead pig for transporting radioactive materials.
Glass container to fit pig.
30-in remote tongs with jaws to fit glass container.
30-in remote tongs to fit 250-ml beaker.
250-ml beaker.
50-ml beaker.
200 ml "dye" solution. (Any bright dye is acceptable.)
Plastic tray.
Absorbent liner for plastic tray.
30-in remote pipetter.
2-ml pipette.
Mirror for reflective viewing.
Closed-circuit TV camera, cables, and monitor.
Stainless steel planchets, 25 mm in diameter.
10-ml volumetric flask.
0.1 μCi of ^{90}Sr/^{90}Y in 5 ml solution.
1 μCi of ^{137}Cs in 1 ml solution.
100-μl Eppendorf pipette and tips.
2-μl Eppendorf pipette and tips.
10 μCi ^{60}Co in 1 ml solution.
Evaporation apparatus assembly.
Silicone grease.
4-cm flat aluminum planchets.
2.5-cm diameter masking tape discs.
Fume hood.
GM survey meter.
12 lead bricks.
Heat lamp equipped with sample spinner.

PROCEDURES

PROCEDURE 1: TONG MANIPULATIONS

This procedure is designed to develop skill in the use of remote-handling tongs.

1. Place glass container that simulates an isotope container into lead transport pig.
2. Place lead transport pig inside a fume hood.
3. Using 30-inch tongs, reach into pig; remove glass container; place it beside lead pig.
4. With a GM survey meter, check jaws of tongs for contamination.
5. Pick up glass container next to pig with 30-inch tongs.
6. Place glass container in lead transport pig.
7. With a GM survey meter, check jaws of tongs for contamination.
8. Repeat procedure at least 4 more times, for a total of 5 times.

PROCEDURE 2: REMOTE PIPETTING

1. Place a plastic tray and tray liner in a fume hood.
2. Place a 250-ml beaker containing 200 ml of a dye solution on liner in tray.
3. Place a 50-ml beaker containing 20 ml of water next to beaker of dye.
4. Connect a 2-ml pipette to remote pipetter.
5. Transfer 2 ml of dye solution to 50-ml beaker. Be sure to rinse out pipette to ensure quantitative transfer of material from pipette.

6. Repeat at least 4 more times, for a total of 5 times. Be sure to use clean water in 50-ml beaker each time.

PROCEDURE 3: MIRROR VIEWING

1. Place lead pig and glass container in fume hood as in Procedure 1.
2. Erect a barrier wall of lead bricks at entrance of fume hood. The wall should be at least 2 bricks wide by 4 bricks high (i.e., 41 cm wide by 40 cm high).
3. Position a mirror in fume hood in such a manner as to permit reflected viewing of both pig and glass container.
4. Perform same manipulations as in Procedure 1, using mirror viewing instead of direct viewing.

PROCEDURE 4: MIRROR VIEWING

1. Place plastic tray, fresh liner, beakers and dye in fume hood as in Procedure 2.
2. Place mirror and barrier wall in fume hood as in Procedure 3.
3. Repeat operations of Procedure 2, using mirror viewing instead of direct viewing.

PROCEDURE 5: VIDEO VIEWING

1. Remove viewing mirror from fume hood.

2. Install a closed-circuit television camera and monitor for viewing inside hood.
3. Repeat activities of Procedures 1 and 2, using video viewing instead of direct or mirror viewing.

PROCEDURE 6: PLANCHET PREPARATION

1. Place a clean absorbent pad on plastic tray in fume hood.
2. Place 250-ml beaker with dye solution on absorbent pad.
3. Place 5 planchets near beaker.
4. Place a clean 100-μl tip on the 100-μl Eppendorf pipette.
5. Place 100 μl of dye solution on each of the 5 planchets.
6. Place planchets on sample spinner under infrared lamp to dry.
7. Inspect absorbent pad for spilled drops of dye. Record in Data Table 20-1-1 the number of spots.
8. Repeat Steps 1 through 6, substituting a solution that contains 0.1 μCi of $^{90}Sr/^{90}Y$ in 5 ml of solution for the dye solution.
9. Take a 5-minute background count with a GM scaler.
10. Take a 3-minute count of each planchet.
11. Record data in Data Table 20-1-2.
12. Calculate average count rate, using all 5 samples. Record in Data Table 20-1-2.

PROCEDURE 7: ACTIVITY PER UNIT VOLUME DILUTION

Given 5 ml of a solution containing 1 μCi/ml of ^{137}Cs, prepare a 10-ml solution containing 0.01 μCi/ml.

1. Calculate required total activity in final dilution.
2. Calculate volume of original solution that contains required total activity.
3. Fill 10-ml volumetric flask approximately 2/3 full with water.
4. Transfer calculated volume of original solution containing required total activity from original solution to water in volumetric flask.
5. Finish filling volumetric flask to the mark with water.
6. Mix solution thoroughly.
7. Transfer solution to a clean, stoppered bottle; label: 10 ml, 0.01 μCi/ml, ^{137}Cs, (date), (name of person who prepared solution).
8. Transfer 1 ml of solution to a 2.5-cm stainless steel planchet; evaporate to dryness.
9. Assay on a single-channel pulse-height analyzer adjusted to count the 0.662-MeV gamma from ^{137}Ba.
10. Record all data in Data Table 20-1-3.

PROCEDURE 8: KNOWN DILUTION

Given 1 ml of ^{60}Co with an activity of 10 μCi, prepare a dilution of 1:5000. NOTE: The entire 1 ml of the original solution need not be used.

1. Select a convenient final volume (i.e., 10 ml).
2. Calculate volume of original solution required to give a dilution of 1:5000 when diluted to 10 ml.

3. Fill 10-ml volumetric flask approximately 2/3 full of water.
4. Transfer required volume of original solution to water in volumetric flask.
5. Finish filling volumetric flask to the mark with water.
6. Mix solution thoroughly.
7. Transfer solution to a clean, stoppered container; label the container.
8. Transfer 100 µl of the solution to a flat planchet, dry, and assay in a Beta counter.
9. Record all data in Data Table 20-1-4.

PROCEDURE 9: LIQUID SAMPLE CONCENTRATION

1. Transfer remainder of the 1 ml of ^{60}Co solution to 1 liter of water.
2. Mix solution thoroughly.
3. Transfer 1 ml of the solution to a planchet; evaporate to dryness.
4. Place a 2.5-cm disc of masking tape on center of one side of a 4-cm aluminum flat planchet.
5. Coat uncovered surfaces of the planchet with silicone grease.
6. Remove masking tape from planchet.
7. Coat inside surfaces of the evaporator plate with silicone grease.
8. Place planchet in the plug for the evaporation unit, with the uncovered spot to the outside. Install plug in evaporator.
9. Adjust variac for approximately 90 volts.

10. Transfer remainder of the liter of solution that contains ^{60}Co to the evaporator.
11. Evaporate solution to dryness.
12. Assay activity of the 4-cm planchet for total activity and activity per ml of original solution.
13. Assay activity in the 2.5-cm planchet to determine activity per ml of original solution.
14. Record all data in Data Table 20-1-5.

DATA TABLES

DATA TABLE 20-1-1

Number of dye spots ______________________________

DATA TABLE 20-1-2

Planchet	Gross Counts for 3 min.	Gross cpm	Background cpm	Net cpm

DATA TABLE 20-1-3

Activity required in final dilution ____________________

Volume of original solution required ____________________

ASSAY

Volume of Sample	Gross cpm	Background cpm	Net cpm	Counter Efficiency	Activity µCi/ml

DATA TABLE 20-1-4

Sample Volume	Gross cpm	Background cpm	Net cpm	Counter Efficiency	Activity μCi/ml

DATA TABLE 20-1-5

Sample Volume	Gross cpm	Background cpm	Net cpm	Counter Efficiency	Activity μCi/ml

REFERENCE MATERIALS

Chase, Grafton D. and Rabinowitz, Joseph L. *Principles of Radioisotope Methodology*. 3rd ed. Minneapolis, MN: Burgess Publishing Co., 1967.

Overman, Ralph T. and Clark, Herbert M. *Radioisotope Techniques*. NY: McGraw-Hill Book Co., 1960.

TEST

1. Sketch and discuss the procedure for using a 2-ml transfer pipette.

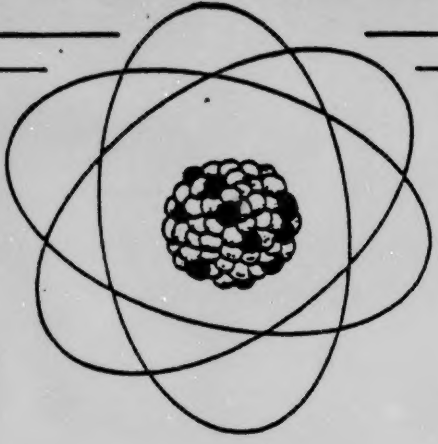
2. List four general rules for any chemical laboratory.

3. List safety precautions to be observed when radioactive materials are being used.

4. List four rules to be observed during the operation of an analytical balance.

5. When may disposable laboratory ware be used?

6. Write a procedure for the preparation of a 5-ml sample of ^{137}Cs containing an activity of 0.25 μCi/ml when given a sample that contains 3 ml having an activity of 7 μCi/ml.



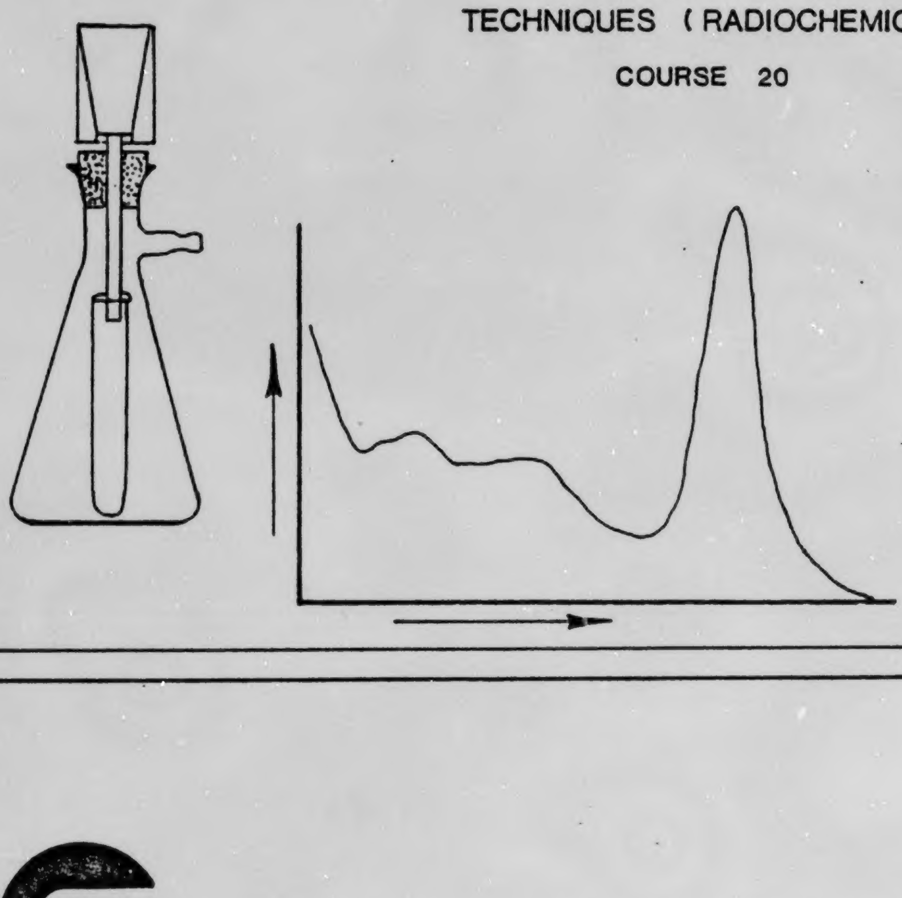
NUCLEAR TECHNOLOGY

MODULE 20 - 2

RADIATION SAMPLE PREPARATION - SOLID

RADIATION MONITORING
TECHNIQUES (RADIOCHEMICAL)

COURSE 20



CENTER FOR OCCUPATIONAL RESEARCH AND DEVELOPMENT

COURSE MODULES

COURSE 20 RADIATION MONITORING TECHNIQUES (RADIOCHEMICAL)

MODULE 20-1	Radiation Sample Preparation – Liquid
⇨ MODULE 20-2	Radiation Sample Preparation – Solid
MODULE 20-3	Air Monitoring Techniques – Particulate
MODULE 20-4	Air Monitoring Techniques – Gaseous
MODULE 20-5	Water Monitoring Techniques – Dissolved Solids
MODULE 20-6	Water Monitoring Techniques – Suspended Solids
MODULE 20-7	Radiation Monitoring of Biological Samples

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INTRODUCTION

Radioactive samples must be prepared according to defined practices if they are to meet specified limits and provide useful and reproducible data. The technician must consider such things as the physical state of the sample, its chemical properties, and its radiation properties. Still other points that must be considered include the type of detector to be used and its characteristics.

Before discussion of radioactive source preparation, this module will include some the safety aspects to be observed in the radiochemical laboratory.

This module discusses the preparation of solid radioactive sources. Methods of preparation discussed include direct evaporation, filtration, electrodeposition, and dry powders. Also explained are sample drying and mounting, specific activity, and extended source geometry.

MODULE PREREQUISITES

The student should have completed Course 1, "Radiation Physics"; and Course 11, "Radiation Detection and Measurement."

OBJECTIVES

Upon completion of this module, the student should be able to:

1. List safety precautions that the technician must observe when working in a radiochemical laboratory.
2. Prepare a card mount of a radioactive material.
3. Prepare at least three different planchet sources of a radioactive material.
4. Prepare a split source of radioactive material for resolving time determination.
5. Calculate the geometry factors for an extended source.

SUBJECT MATTER

RADIOCHEM LAB SAFETY

The maximum allowable radiation dosage for workers using radioactive materials has been set by the Nuclear Regulatory Commission (NRC) and the several Agreement States. Each time the numbers have been revised, the revision has been downward. Current regulations allow a "rule-of-thumb" guide of exposure of 100 millirem per week. However, the general consensus is that no individual should receive any larger dose than is necessary.

Considering this, most laboratories urge their employees to limit their radiation exposure to no more than is absolutely required. As a means to this, most laboratories prepare rules for safe operation and require that their employees conform to these laboratory safety regulations. The following list of regulations is typical of those found in most organizations in which radiochemicals are used.

RADIATION CHEMICAL LABORATORY SAFETY REGULATIONS

1. When on duty, workers must wear personnel monitoring devices at all times.
2. Personnel who have open wounds are not permitted in contaminated areas or in areas in which unsealed radioactive materials are used.
3. Eating, drinking, smoking, or the application of cosmetics is prohibited in all areas in which corrosives, poisons, or unsealed radioactive materials are in use.

4. Pipetting by mouth is strictly prohibited.
5. Fume-hood exhaust fans must be operating when the hoods are in use.
6. Wear appropriate anti-contamination clothing when in the radiochemistry laboratory.
7. Remove anti-contamination clothing when leaving the radiochemistry laboratory.
8. Monitor radiation levels of hands and feet when leaving the radiochemistry laboratory.
9. If the hands or feet are contaminated, or contamination of another area of the body or of equipment is suspected, notify Health Physics, and proceed with routine decontamination procedures.
10. Place radioactive liquid waste in the containers provided. NEVER pour into a standard drain.
11. Radioactive solid wastes and contaminated materials must be placed in containers labeled "radioactive solid waste."
12. Be sure that all required records are completed.
13. Normally, radioactive materials and contaminated items are to be retained within the laboratory.
14. Should a spillage occur, the following procedure is to be followed:
    a. Notify Health Physics.
    b. Spilled liquids should be blotted up with disposable wipes. (Rubber gloves must be worn.)
    c. Disposable materials contaminated by the spill should be placed in a container labeled "radioactive solid waste."
    d. The contaminated area should be decontaminated.
    e. Survey the decontaminated area to determine residual contamination.

f. Mark any residual contamination to identify location, type, and amount of activity.

Usually, close attention to good housekeeping, attention to detail, and good laboratory techniques will be all that is required for a lifetime of safe laboratory activity.

SOLID SOURCES

Many radionuclides can be counted satisfactorily as solid sources with the simplest types of equipment. Preparation of these solid sources may be simple or complex, but three general steps usually are involved. These steps are —

- Preparation of the source mount, backing, or support.
- Depositing of the radioactive material.
- Sealing of the deposited material.

Because the source mounts usually are prepared commercially and it may be desirable not to seal the deposited material in the mount, this section will be concerned principally with the deposition of the radioactive materials and the treatment of the deposit. Generally, samples must be uniform, physically, chemically, and geometrically. These requirements are especially stringent for weak beta emitters.

DIRECT EVAPORATION

One of the more common methods of deposition is the evaporation of a solution containing the radioactive material in a nonvolatile form dissolved in a volatile solvent. This technique is widely employed, especially for calibration standards, where the specific activity is high and there is physically very little mass of material in solution.

The actual procedure is quite simple; however, refinements may become complicated. Usually, a small volume of the liquid is placed on a mount and evaporated to dryness by an infrared lamp. Care must be taken to obtain uniform coverage of a specific area and to prevent sputtering during drying. Creeping of the sample may occur, especially with volatile liquids. This can be minimized by the addition of a less volatile liquid before transfer to the mount. There is a tendency for an excessive deposit to form around the edges of cup planchets. Deposit can be minimized either by using ringed planchets or, if the samples are dried on a hot plate, by supporting the planchet on a hollow metal cylinder. This procedure selectively heats the edges and causes the solids to be drawn to the center of the planchet. There are also commerical devices that provide a slowly revolving turntable upon which the planchet is positioned. The liquid sample then is applied slowly, while a continuous source of heat rapidly evaporates the solvent.

The major factor in these operations is the accuracy of pipetting. Quite a variety of commercial pipettors is available, and specific operations vary with the manufacturer. It is extremely important that the student or technician obtains as much practice as possible with the pipettors available in order to become proficient with their use. Remember that pipetting in a radiochemical laboratory is NEVER done by mouth.

FILTRATION

Solid counting samples also are prepared from precipitates or slurries. Many filtration devices have been developed for the purpose of quantitative collection of precipitates for radioassay. Two of the most significant developments in filtration are microporous filtration materials and fibrous glass filters. Additionally, the development of modern filtration devices almost has eliminated the use of sintered-glass Gooch crucibles. Presently, filtered sample preparation offers excellent reproducibility, allows the area and thickness of the sample to be controlled within very narrow limits, and provides for easy comparison between large numbers of samples.

Filtration apparatus for radioactive precipitates should meet the following requirements:

- The filter medium should serve as the source mount.
- The filter should retain the precipitate with miniumum loss into the filtrate.
- The filter should give an even layer of the precipitate.
- The deposited material should be well defined, centered, and reproducible.
- The filter medium should be easily removable from the apparatus without disturbing the deposited material.
- The apparatus should not leak.
- The apparatus should not be attacked by common reagents.
- The design should permit washing of the precipitate from the walls onto the filter medium.
- The apparatus should be easily decontaminated.

Figure 20-2-1 illustrates a filter assembly for the collection of precipitates on filter paper.

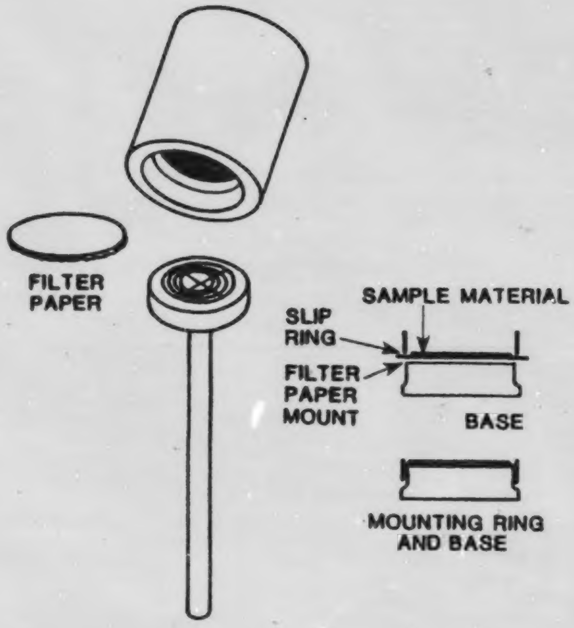


Figure 20-2-1. Filter assembly for collection of precipitates on filter paper.

Care must be taken to assure that the cake of precipitate on the filter medium does not crack either during filtration or subsequent drying operations. The following procedures will aid in prevention of filter cake cracking:

1. Always filter slowly. Adjust the applied pressure difference by bleeding or bypassing the vacuum to obtain the proper filter rate for each substance filtered. Use a gage or manometer to monitor and to maintain vacuum levels for reproducibility.
2. When needed, add a dilute binder, such as collodion, before drying.
3. Dry slowly with suction applied, either with or without a heat lamp over the filtration apparatus. Drying time can be reduced by rinsing with alcohol and then acetone if these solvents will not attack the precipitate.

4. After the filtration is complete, gently admit air into the filtration flask.

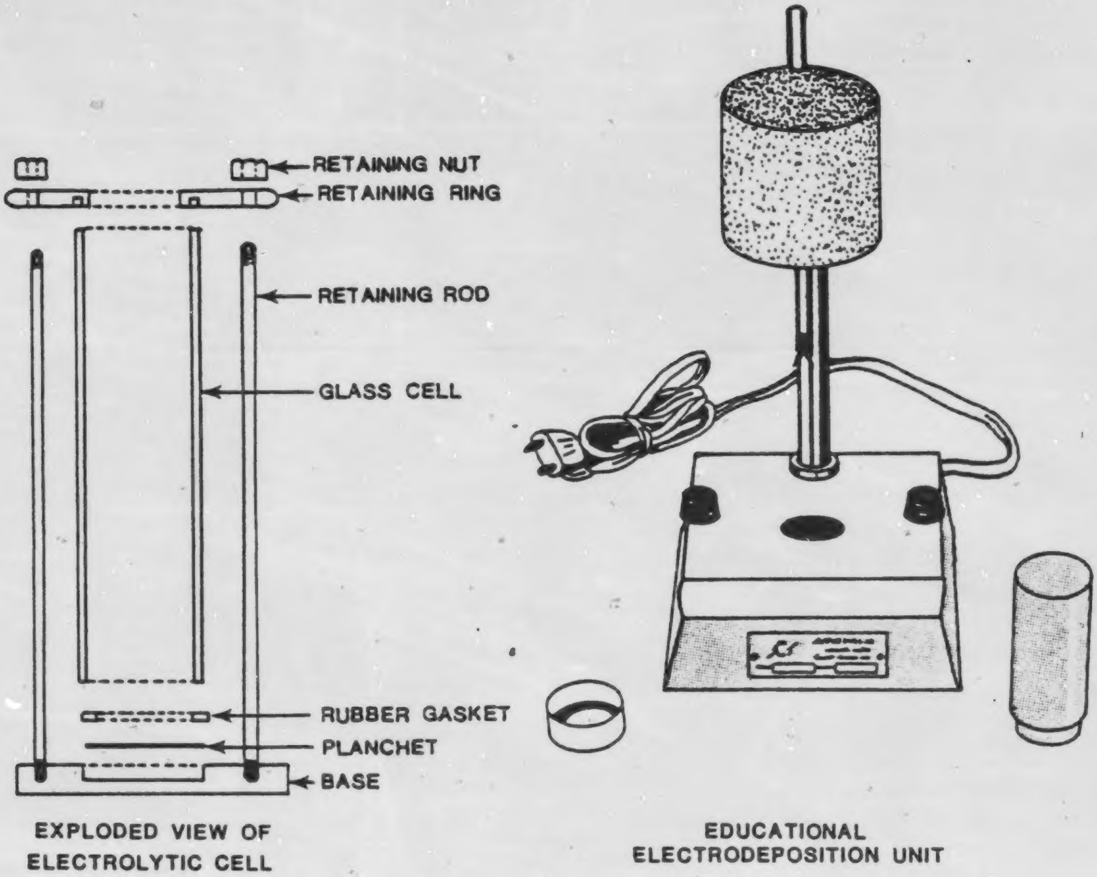
There is one other consideration when preparing filtered samples. When mass of the deposited material is to be determined, the filter medium should be pretreated with all the wash solutions to be used in the preparation of the sample and then dried in the same manner as the sample before the initial mass of the filter media is determined.

Slurry samples may be transferred directly to planchets with a transfer pipette, but most workers find it more convenient to add a bit more solvent and to transfer to a filter.

ELECTRODEPOSITION

Solid samples of some elements can be prepared conveniently for counting by electrodeposition on a conducting surface. Usually, the active material is deposited in elemental form, but a few elements are deposited as oxides. The major advantage of this method is the preparation of very thin films of uniform thickness, which are particularly useful for elements that decay by electron capture, by weak beta emission, or by alpha emission.

Several commercial units are available for general work. The apparatus typically consists of a leak-proof cell formed by a glass cylinder, a rubber gasket, and a flat metal disk that serves as the cathode and as the bottom of the cell. The anode usually is a rotating spiral of platinum wire. Normally, a few milligrams of an isotopic carrier are added before electrodeposition to ensure good deposition of the plated material. Figure 20-2-2 illustrates an electrodeposition apparatus.


RETAINING NUT
RETAINING RING
RETAINING ROD
GLASS CELL
RUBBER GASKET
PLANCHET
BASE

EXPLODED VIEW OF ELECTROLYTIC CELL

EDUCATIONAL ELECTRODEPOSITION UNIT

Figure 20-2-2. Electrodeposition apparatus.

The procedures are seldom the same as those for commerical electroplating, although the variables to be controlled are identical. Optimum conditions for each of the following eight variables must be maintained to obtain a good quality, adherent deposit, and all vary for each element.

- Applied voltage.
- Current density.
- Distance between electrodes.

- Shape and size of the rotating electrode.
- Speed of rotation of the rotating electrode.
- Agitation of the solution.
- Concentration of the solution.
- Temperature.

Sources can be prepared from some elements by the related process of chemical displacement with a more active element. However, this technique is very limited.

The major disadvantage of electrodeposition is the time and labor required as compared with some of the direct methods.

DRY POWDERS

Dry powders can be pressed into a planchet with a spatula or formed into briquets by a laboratory press. In many cases, the dry powder simply can be placed in a planchet for counting. These methods, however, are unacceptable for samples that are required to be very thin. When a powder sample must be used, it is advisable to apply a binder of 0.5% collodion to prevent spillage and contamination of the counting apparatus. Usually, powder samples are limited to thin samples of strong beta or gamma emitters.

SAMPLE DRYING

Sample drying involves the removal of some solvent from the sample. Normally, the material to be removed is water, but it can be any of a number of organic solvents.

INFRARED LAMPS

Probably the most useful apparatus in the modern sample-preparation laboratory is an infrared lamp. This lamp provides a method of applying an even, easily controlled heat to samples for drying. There are commercial devices available that will rotate a sample continuously while it is drying. Rotation provides a more uniform application of heat and, subsequently, a more uniform drying of the sample.

Two precautions must be observed when a sample is dried: they apply to all methods of drying that involve heat. First, the sample should not be dried too rapidly. A sample dried too rapidly will crack or curdle and will affect the sample geometry in such a way as to render the sample almost useless for good quantitative counting.

Second, the sample should not be dried too much after the solvent has evaporated. Excessive heating could cause the sample to change chemical composition or could volatilize a portion of the sample. Both of these precautions notwithstanding, infrared drying presently is considered the "best" procedure for solid samples.

MOUNTING MATERIALS

Selection of a source mounting material is very important in the preparation of radioactive solid sources. Resistance of the material to chemical attack, beta backscatter characteristics, resistance to heat, and cost are factors that must be considered during the selection of a mounting material. Table 20-2-1 lists some of the common mounting materials and their resistance to attack.

TABLE 20-2-1
CHEMICAL RESISTANCE OF SOME MOUNTING MATERIALS

Filter Paper	Attacked by strong acids and bases
Glass Fiber Paper	Attacked by NaOH and HF
Cellophane Tape	Attacked by organic solvents and conc. H_2SO_4: useful for powder sources
Aluminum	Attacked by acids and strong caustics
Copper	Attacked by HNO_3; useful for electrodeposition
Nickel Plated Steel	Attacked by conc. acids
Stainless Steel	Attacked by HCl and HF
Plantinum	Attacked by aqua regia; useful for electrodeposition and ignition
Polyethylene	Relatively inert; useful for HF
Polystyrene	Attacked by organic solvents
Teflon	Relatively inert; useful for HF

Filter papers and other organic materials are sensitive to heat; therefore, care must be taken not to overheat them during drying. This is also true for many organic samples that might be deposited as thin films. In some cases, thermal decomposition will occur at temperatures well below 100°C.

Additionally, consideration must be given to the mounting material when samples are being prepared for very low levels of counting. Naturally-occurring radioisotopes appear in many products; for instance, glass usually has sufficient ^{40}K to provide count rates of up to several counts per minute during counting of beta radiation. Many metals are contaminated with sufficient uranium, radium, and their daughters to affect appreciably the alpha and gamma count rates. Therefore, the conscientious technician must routinely check the mounting materials for contamination.

SPECIFIC ACTIVITY

The strictest definition of "specific activity" is the ratio of the number of radioactive atoms to the total number of isotopic atoms; that is, the total radioactivity of a given isotope per gram of the radioactive isotope. Additional definitions, although less correct, are in common use.

One definition is the element specific activity. This is defined as the total radioactivity of a given isotope per gram of the element. Care must be exercised not to confuse this with the strict definition.

Labeled compounds usually are identified by their compound specific activity — the total activity of a given isotope per gram of a compound. This seems to be the definition in most common use. With a little thought, one should be able to shift from one definition to the other.

Another definition involves the activity per mole of material, and still another definition involves activity per unit volume. All of these terms are acceptable to the radiation chemist and health physicist. However, in preparation of solid samples for counting, it is desirable to consider activity per unit mass of material, which, when properly applied, will yield a correction counting factor called "sample self-absorption." This is of paramount importance in the case of weak beta emitters, but corrections also must be made for high-energy emitters. Gamma radiation is so penetrating that the sample self-absorption effect frequently is negligible; therefore, the following discussion will be concerned with beta emitters.

DILUTION TO CONSTANT MASS

One technique utilized in the preparation of solid samples is dilution to a constant mass. This procedure requires addition of inactive material, usually of the same chemical composition as the radioactive material, to increase the total mass to a constant value. The sample then is mixed thoroughly, and the sample is counted.

The disadvantages of this technique are obvious. Changing of the specific activity of a sample by addition of inert material can interfere substantially with any additional tests that may be required. Likewise, addition of inert material will reduce the activity, thereby increasing required counting times. In some cases, this reduction can render radioassay procedures completely useless. Figure 20-2-3 illustrates the effect of changed concentration on count rate when the sample is maintained at a constant mass.

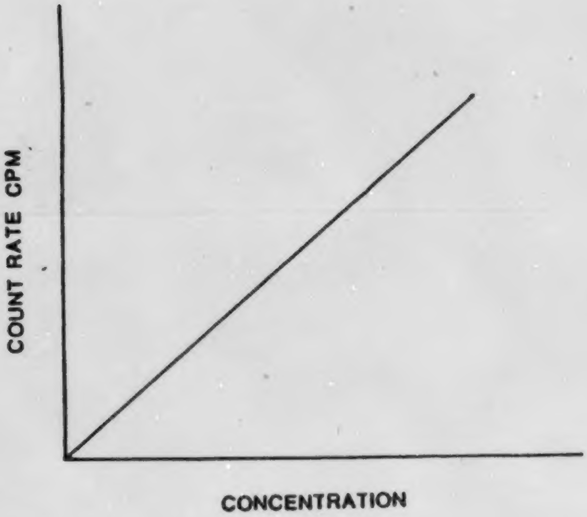


Figure 20-2-3. Observed activity for constant mass samples.

CONSTANT TOTAL ACTIVITY

Another technique in preparation of solid samples for counting involves comparison of samples that have the same total activity but different masses. Figure 20-2-4 illustrates the change in count rate observed as the mass is increased. The indication is that, at a constant total activity, the observed count rate is approximately inversely proportional to the mass of the sample and theoretically would approach zero at infinite dilution.

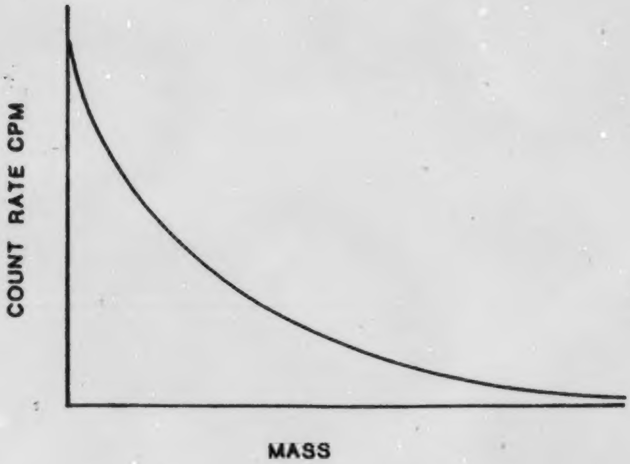


Figure 20-2-4. Observed activity relation at constant total activity.

CONSTANT SPECIFIC ACTIVITY

The term "constant specific activity" refers to samples that contain the same activity per unit mass. When samples contain a constant specific activity, doubling of the mass will double the total activity of the sample. However, because of sample self absorption, the observed count rate will not be doubled. Figure 20-2-5 illustrates the effect of increased sample thickness on the count rate of a sample of constant specific activity.

As thickness of the source is increased, the observed count rate is increased, but not directly in proportion. This process continues until the observed rate rises to a constant maximum. The constant maximum, or "infinite thickness counting rate," is achieved when particles from the lowest portion of the sample are absorbed before they reach the surface of the sample. Thus, the infinitely thick sample is defined.

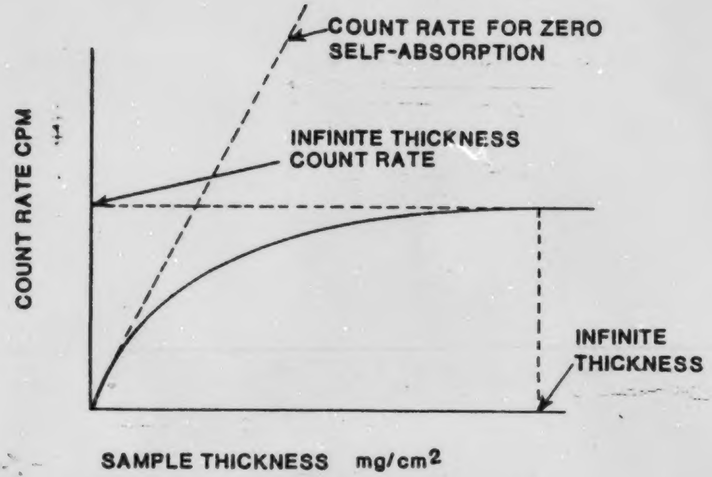


Figure 20-2-5. Constant specific activity self absorption.

MATHEMATICAL CORRECTION

Sample self absorption can be corrected mathematically if the beta energy and the density thickness of the sample are known. The formula for correcting the observed count rate to the true count rate is shown in Equation 20-2-1.

$$R_t = R_o \frac{0.693 \frac{x}{d_{\frac{1}{2}}}}{1 - (\frac{1}{2})^{x/d_{\frac{1}{2}}}} \qquad \text{Equation 20-2-1 (Equation = Eq.)}$$

where:

R_t = true count rate.
R_o = observed count rate.
x = density thickness of the sample in mg/cm².
$d_{\frac{1}{2}}$ = beta half-thickness in mg/cm².

EXAMPLE

Given: A sample of ^{14}C barium carbonate found to have a count rate of 962 cpm and a density thickness of 32 mg/cm^2.

Find: The true count rate.

Solution: From the Radiological Health Handbook, the beta energy of ^{14}C is 0.156 MeV, and the half-thickness for 0.156 MeV beta particles is 2.2 mg/cm^2. Use Equation 20-2-1:

$$R_t = R_o \frac{0.693 \frac{x}{d_{\frac{1}{2}}}}{1 - (\frac{1}{2})^{x/d_{\frac{1}{2}}}}$$

$$= 962 \text{ cpm } \frac{0.693 \; 32 \text{ mg/cm}^2/2.2 \text{ mg/cm}^2}{1 - (0.5)^{32/2.2}}$$

$$= 962 \text{ cpm } \frac{0.693 \quad 14.5}{1 - 0.000043}$$

$$R_t = 9667 \text{ cpm.}$$

As seen from the above example, weak beta emitters must be corrected for sample self absorption. However, there are other procedures that can correct for self absorption without direct calculation.

INFINITELY THIN SAMPLES

One method of dealing with sample self absorption simply is to prevent it from occurring. Solid samples frequently are prepared either by evaporation of a solution to dryness or by collection of a precipitate on a filter, after which it is dried. When solutions are evaporated to dryness, the quantity of the residue often can be assumed to be negligible. This is particularly true in cases involving materials of high specific activity. Samples such as this are considered to be "infinitely thin."

Collected samples and dried precipitates rarely are infinitely thin. Usually, they fall somewhere between infinitely thin and infinitely thick.

INTERMEDIATE THICKNESS SAMPLES

Samples neither infinitely thick nor infinitely thin must be adjusted to some standard of mass or thickness in order to provide useful data. The preceding method of calculation may be used or the corrections may be determined experimentally by the procedure that follows.

1. Prepare at least four samples of increasing density thickness. This usually is accomplished by use of multiples of the original sample.
2. From the observed activity of the four samples, determine the activity and thickness of an infinitely thick sample by graphing the activity of each of the four samples versus the density thickness of each (cpm versus mg/cm^2).

3. Prepare a second graph of the activity of the four samples as cpm/mg versus density thickness.
4. Extrapolate the second graph to zero thickness to determine the specific activity of an infinitely thin sample.
5. Calculate the self absorption correction factor:

$$F_s = \frac{\text{specific activity for infinite thickness}}{\text{specific activity for infinitely thin sample}} \qquad \text{Eq. 20-2-2}$$

Once the self absorption factor has been determined, the corrected count rate can be calculated from Equation 20-2-3.

$$R_t = \frac{R_o}{F_s} \qquad \text{Eq. 20-2-3}$$

where:

R_t = corrected count rate.
R_o = observed count rate.
F_s = self absorption correction factor.

One of the Procedures at the end of this module involves the determination of a self absorption correction factor.

EXTENDED SOURCE GEOMETRY

Radioactive samples that are not point sources are extended sources. Most of the samples routinely encountered will be large enough to consider as extended sources. The sources normally should not be larger than 1/2 the diameter of the detector, although at times they may be of equal diameter. Equation 20-2-4 is a formula for calculation of the geometry factor for a point source.

$$G = 0.50 \cdot 1 - \frac{F}{(1 + b^2/a^2)^{\frac{1}{2}}} \qquad \text{Eq. 20-2-4}$$

where:

G = geometry factor of a point source.
F = 1.
a = distance from point source to detector.
b = radius of the detector.

The calculation of extended source geometry, involving plane-to-plane locations, considerably changes the value of F. Figure 20-2-6 illustrates the locations of the variables in physical perspective. The value for F becomes —

$$F = 1 + 0.375J - 0.3125JK + 0.546875J^2 + 0.2734375JK^2 - 1.23046875J^2K + 1.12792968J^3.$$

where:

$$J = \frac{b^2}{a^2 + b^2} \times \frac{c^2}{a^2 + b^2}.$$

$$K = \frac{a^2}{a^2 + b^2}.$$

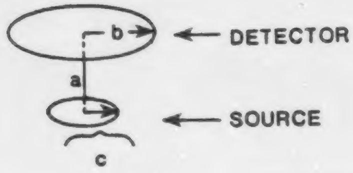


Figure 20-2-6. Location of the variables a, b, and c.

Equation 20-2-4 then takes the form of Equation 20-2-5.

$$G = 0.50 \quad 1 - \frac{1 + 0.375J - 0.3125JK + 0.546875J^2 + 0.2734375JK^2 - 1.23046875J^2K + 1.12702968J^3}{1 + (\frac{b^2}{a^2})^{\frac{1}{2}}}$$

Eq. 20-2-5

The following example illustrates the difference in geometry factor for a point source and extended source of 2.5 cm diameter.

EXAMPLE

Given: Detector diameter, 2.9 cm; source to detector distance, 2.6 cm; and source diameter, point and 2.5 cm.

Find: The geometry factor for each source.

Solution: Point source, Equation 20-2-4:

$$G = 0.50 \quad 1 - \frac{F}{\left(1 + \frac{b^2}{a^2}\right)^{\frac{1}{2}}}$$

$$= 0.50 \quad 1 - \frac{1}{1 + \left(\frac{1.45^2}{2.6^2}\right)^{\frac{1}{2}}}$$

$$G = 0.06.$$

Extended source, Equation 20-2-5:

$$G = 0.50 \quad 1 - \frac{1 + 0.375J - 0.3125JK + 0.546875J^2 + 0.2734375JK^2 - 1.23046875J^2K + 1.12702968J^3}{1 + (\frac{b^2}{a^2})^{\frac{1}{2}}}$$

where:

$$J = \frac{b^2}{a^2 + b^2} \times \frac{c^2}{a^2 + b^2} = \frac{2.1}{8.86} \times \frac{1.56}{8.86} = 0.042.$$

$$K = \frac{a^2}{a^2 + b^2} = \frac{2.76}{8.86} = 0.763$$

$$G = 0.50 \quad 1 = \frac{1 + 0.01575 - 0.01001 + 0.0096 + 0.006686 - 0.001656 + 0.000074}{1.1448}$$

$$G = 0.054.$$

EXERCISES

1. Rewrite the radiation chemical laboratory safety regulations, grouping according to regulations for the prevention of the spread of contamination, regulations for personal safety, and others.
2. Write a procedure for the preparation of a solid source of 1 μCi of ^{60}Co from an original solution of 30 μCi of ^{60}Co contained in 1 ml of water.
3. Write the procedure for the preparation of an uncracked sample of ^{14}C-barium carbonate mounted on a fiberglass filter. The sample should contain 5 μCi of ^{14}C, and the original supplied solution contained 50 μCi of ^{14}C as $NaHCO_3$ in 5 ml of water.

MATERIALS

Card mounts.
Plastic wrap (Saran or similar).
Infrared heat lamp.
Analytical balance.
Calipers.
Glass fiber filter discs.
Vacuum filtration apparatus.
Large test tubes.
Water bath.
Desiccator.
Electrodeposition apparatus.
Copper discs.
Whatman #1 filter paper.

Beta counter.
Gamma counter.
Non-ionic detergent.
95% alcohol.
Barium chloride.
Sodium sulfate.
Ammonium sulfate.
Ammonium hydroxide, concentrated.
Hydrochloric acid, dilute.
Ammonium hyposulphite.
1 μCi ^{60}Co.
10 μCi ^{35}S.
Metal shear.

PROCEDURES

PREPARATION OF A POINT SOURCE

1. Prepare a card mount with plastic wrap taped over the center hole.
2. Transfer 0.1 μCi of ^{35}S from stock solution containing 10 μCi of ^{35}S per ml of solution to the plastic wrap on the card mount. Chemically, the ^{35}S should be Na_2SO_4.
3. Evaporate liquid from the sample with an infrared heat lamp. Be careful not to overheat the sample. DO NOT COVER THE SAMPLE WITH PLASTIC WRAP.
4. Label sample with the isotope, quantity, date, and student's name.

5. Determine the count rate for the point source; record in Data Table 20-2-1.
6. Place sample in a sample container; store in the isotope cabinet for future reference.

PREPARATION OF GLASS FIBER FILTER MOUNTS OF ^{35}S AT CONSTANT TOTAL ACTIVITY

1. Prepare 6 card mounts to hold glass fiber filters.
2. Rinse 6 glass fiber filters with water, then with 95% alcohol; dry and weigh each filter.
3. Dry and weigh each filter a second time.
4. If there is more than 0.2 mg weight difference between the first and second weighings, repeat drying and weighing until difference is within 0.2 mg. Store the dried and weighed filters in a desiccator.
5. Measure inside diameter of the filter chimney.
6. Calculate area of the precipitate that will be deposited on the glass fiber filter.
7. Prepare 6 test tubes, each containing approximately 5 ml of water.
8. Using the area of precipitate deposit as calculated in Step 6 above, calculate the mass of $BaSO_4$ required to produce deposits of approximately 2.5, 5, 10, 20, 30, and 40 mg/cm^2. A solution that is 0.1 N in $SO_4^=$ will yield 16.7 mg of $BaSO_4$ per ml.
9. Transfer 0.1 μCi of ^{35}S as $SO_4^=$ to each of 6 of the test tubes.
10. Transfer quantities of 0.1 N $SO_4^=$ solution to the test tubes, as calculated in Step 8 above.

11. Add 3 drops of dilute HCl and a drop of non-ionic detergent to each test tube.
12. Add slightly more than an equal volume of 0.1 N $BaCl_2$ to each of the test tubes, as in Step 10 above.
13. Place on a boiling-water bath for 2 minutes.
14. Allow to cool for 5 minutes.
15. Add 1 ml to 0.1 N $BaCl_2$ solution to each test tube, and allow to stand an additional 5 minutes.
16. Assemble a filter chimney with a previously weighed glass fiber filter.
17. Transfer precipitate from a test tube to filter apparatus. Be sure to rinse all of the precipitate into the filter chimney.
18. Filter, beginning with gentle suction.
19. Suction the precipitate dry.
20. Rinse the precipitate with 3 to 5 ml of 95% alcohol, and suction dry.
21. Remove the glass fiber filter from the filter apparatus; dry the precipitate on the filter with an infrared lamp.
22. Weigh the sample.
23. Repeat drying and weighing until the weight does not vary more than 0.2 mg between weighings.
24. Repeat Steps 16 through 23 for each of the remaining 5 samples.
25. Mount each weighed sample on a card mount; label with the isotope, amount, weight, date, and student's name.
26. Beta count the samples, record the data in Data Table 20-2-1.
27. Prepare a graph of cpm versus thickness of the sample ing mg/cm^2.
28. Explain the reasons why the graph has this particular shape.

PREPARATION OF GLASS FIBER FILTER MOUNTS OF ^{35}S AT CONSTANT SPECIFIC ACTIVITY

1. Prepare 8 mounts to hold glass fiber filters.
2. Rinse 8 glass fiber filters with water, then with 95% alcohol; dry and weigh each filter.
3. Dry and weigh each filter a second time.
4. If there is more than 0.2 mg weight difference between the first and second weighings, repeat the drying and weighing until the difference is within 0.2 mg.
5. Store the dried and weighed filters in a desiccator.
6. Measure inside diameter of the filter chimney.
7. Calculate the area of the precipitate that will be deposited on the glass fiber filter.
8. Prepare 8 test tubes, each containing approximately 5 ml of water.
9. Prepare 125 ml of 0.1 N Na_2SO_4 containing 5 μCi of ^{35}S.
10. Using the area of the filter, as calculated in Step 7 above, calculate the volume of 0.1 N Na_2SO_4 required to produce deposits of 5, 10, 15, 20, 25, 30, 35, and 40 mg/cm^2 on the glass fiber filters. A solution that is 0.1 N is $SO_4^=$ will produce 16.7 mg of $BaSO_4$ per ml of solution when fully precipitated.
11. Transfer the quantities of 0.1 N Na_2SO_4 solution to the 8 test tubes.
12. Add 3 drops of dilute HCl to each test tube.
13. Heat the test tubes for 2 minutes on a boiling-water bath.
14. Prepare 125 ml of a solution that is 0.12 N $BaCl_2$.
15. Remove test tubes from water bath; add one drop of a non-ionic detergent; then add the same volume of the $BaCl_2$ solution as the Na_2SO_4 solution that was done in Step 11.

16. Allow test tubes to cool for at least 5 minutes.
17. Add 1 more ml of $BaCl_2$ solution to each test tube; allow to stand for an additional 5 minutes or longer.
18. Assemble filter apparatus with a previously weighed glass fiber filter.
19. Transfer contents of one test tube to the filter apparatus, attempting to get as much of the precipitate as possible. (To obtain every last bit of the precipitate is unnecessary, however.)
20. Filter, beginning with gentle suction.
21. Rinse filter cake 5 times with hot distilled water, using suction.
22. Rinse filter cake 2 times with 95% alcohol, using suction.
23. Gently release suction.
24. Carefully remove glass fiber filter from filter apparatus; dry the precipitate on the filter, using an infrared lamp.
25. Repeat drying and weighing until the weight does not vary more than 0.2 mg between weighings.
26. Mount the glass fiber filter, and precipitate; label with the isotope, amount, weight, date, and student's name; place in a desiccator.
27. Repeat Steps 18 through 26 for each sample.
28. Beta count the samples; record all data on Data Table 20-2-2.

ELECTRODEPOSITION

1. Assemble the electrodeposition apparatus, using a clean copper planchet (disc).
2. Prepare an electrolyte solution containing 10 g ammonium sulfate, 18 ml of concentrated ammonium hydroxide, and 0.5 g of ammonium hypophosphite per 100 ml of solution.
3. Measure a quantity of the electrolyte sufficient for the electrodeposition apparatus; add enough cobalt sulfate or nitrate to provide approximately 0.3 mg of cobalt carrier per ml of solution.
4. Add 1 μCi of ^{60}Co to the solution.
5. Place the electrolyte solution containing ^{60}Co into the electrodeposition apparatus.
6. Electroplate for 6 to 8 hours at 25 mg/cm^2.
7. Remove planchet from the apparatus; rinse and dry the sample.
8. Beta and gamma count the sample.
9. Wipe test active surface of the planchet with a Whatman #1 filter paper.
10. Beta and gamma count the filter paper.
11. Shear the sample in half, using a metal shear, thereby making a split source for resolving time determination.
12. Count each half separately and then together.
13. Record all data in Data Table 20-2-3.

DATA TABLES

DATA TABLE 20-2-1
CONSTANT TOTAL ACTIVITY

Sample	Weight mg	Area cm^2	Density Thickness mg/cm^2	Gross cpm	BG cpm	Net cpm
1						
2						
3						
4						
5						
6						

DATA TABLE 20-2-2
CONSTANT SPECIFIC ACTIVITY

Sample	Weight mg	Area cm^2	Density Thickness mg/cm^2	Gross cpm	BG cpm	Net cpm
1						
2						
3						
4						
5						
6						
7						
8						

DATA TABLE 20-2-3
ELECTRODEPOSITION

Sample	Beta Counts			Gamma Counts		
	Gross cpm	BG cpm	Net cpm	Gross cpm	BG cpm	Net cpm
Disc						
Filter paper						
First half						
Second half						
Both halves						

REFERENCE MATERIALS

Chase, Grafton D. and Rabinowitz, Joseph L. Principles of Radiosotope Methodology. 3rd ed. Minneapolis: Burgess Publishing Co., 1967.

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Faires, R.A. and Parks, B.H. Radioisotope Laboratory Techniques. London: Butterworth & Co. Ltd., 1973.

Nesmeyanov, An. N.; Baranov, V.I.; Zaborenko, K.B.; Rudenko, N.P.; and Priselkov, Yu. A. Handbook of Radiochemical Exercises. NY: The MacMillian Co., 1965.

Overman, R.T. and Clark, H.M. Radioisotope Techniques. NY: McGraw-Hill Book Co., 1960.

TEST

1. Prepare a list of 10 safety precautions that the technician must observe when working in a radiochemical laboratory.

2. Write the procedure for preparation of an infinitely thin, point-source card mount.

3. Write the procedure for preparation of a source from a precipitated source having a constant total activity.

4. Write the procedure for preparation of a glass fiber filter mount from a precipitate having a constant total activity.

5. Write a procedure for preparation of an electrically deposited split source.

6. Why is there a difference between the geometry factor of a point source and the geometry factor of an extended source.

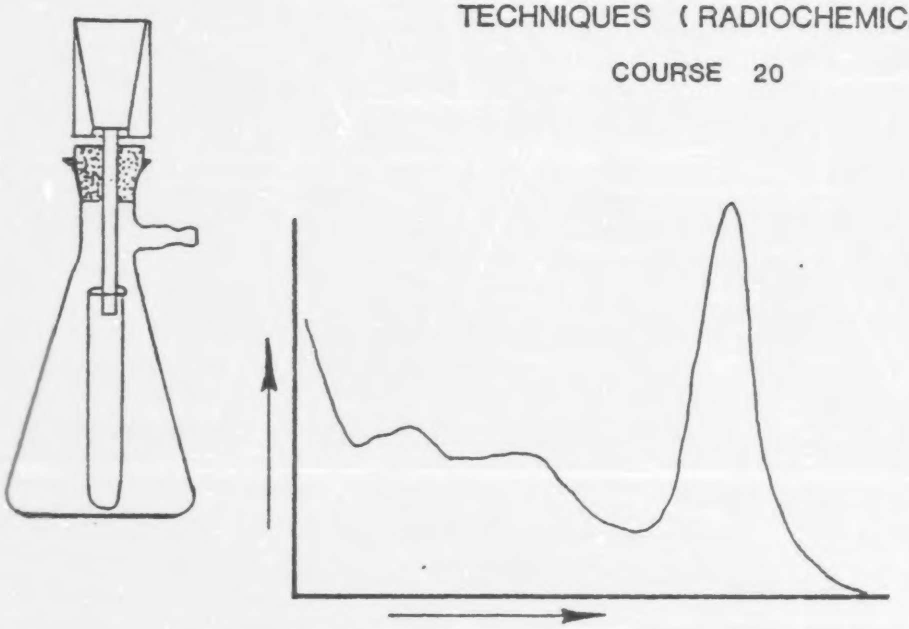
NUCLEAR TECHNOLOGY

MODULE 20 - 3

AIR MONITORING TECHNIQUES - PARTICULATE

RADIATION MONITORING
TECHNIQUES (RADIOCHEMICAL)

COURSE 20



CENTER FOR OCCUPATIONAL RESEARCH AND DEVELOPMENT

COURSE MODULES

COURSE 20 RADIATION MONITORING TECHNIQUES (RADIOCHEMICAL)

MODULE 20-1	Radiation Sample Preparation – Liquid
MODULE 20-2	Radiation Sample Preparation – Solid
⇨ MODULE 20-3	Air Monitoring Techniques – Particulate
MODULE 20-4	Air Monitoring Techniques – Gaseous
MODULE 20-5	Water Monitoring Techniques – Dissolved Solids
MODULE 20-6	Water Monitoring Techniques – Suspended Solids
MODULE 20-7	Radiation Monitoring of Biological Samples

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INTRODUCTION

Inhalation of radioactive materials may cause intense irradiation of tissues of the respiratory tract. The amount of irradiation will depend upon concentration, particle size, solubility, and length of time that an individual is exposed to the contaminating radionuclides.

Contaminants may occur as dusts, fumes, smokes, vapors, mists, or gases. Dusts, fumes, smoke, and mists are particulate matter in the form of solids or liquid droplets. Sampling of these particulates is the important concern of this module.

MODULE PREREQUISITES

The student should have completed Course 1, "Radiation Physics"; Course 11, "Radiation Detection and Measurement"; and Modules 20-1, "Radiation Sample Preparation — Liquid"; and 20-2, "Radiation Sample Preparation — Solid."

OBJECTIVES

Upon completion of this module, the student should be able to:

1. Calculate the alpha and beta-gamma half-life of a background sample.
2. Draw a schematic of a continuous air particulate monitor and label the major components.

3. Calculate activity per unit volume of a given air sample containing a radioactive particulate.
4. Using a portable air sampler, collect a sample, and assess the radioactivity present.

AIR SAMPLING

Air sampling is done to determine presence or absence of contamination in the air environment of a work area or of air to be exhausted to the outside. Additionally, it is done to determine the effectiveness of work procedures or laboratory and equipment design. Sampling for radioactive material in the air falls within this general framework. In this case, the radioactivity is usually the contaminant, and also provides the method of analysis once the sample is collected.

Theoretically, regulations that apply to external radiation dose should apply to internally deposited emitters – the permissible levels then being affected by the sum of both internal and external doses. However desirable such an arrangement would be, the accurate, quantitative dosimetry of internal emitters is difficult. The practical solution to this problem is to hold the environmental concentrations of radionuclides at as low a level as possible. Air samples are one method of confirming that concentrations are held at acceptable levels.

CLASSIFICATION OF CONTAMINANTS

Air contaminants usually are divided into two groups. The first group for consideration is particulates. The second group consists of gases and vapors and will be the subject of Module 20-4, "Air Monitoring Techniques – Gaseous."

Particulate matter includes solid and liquid particles that range down to approximately $10^{-3}\mu$, which is considered to be molecular size. Solids are subdivided into dusts, fumes, and smokes, principally upon the basis of their method of generation. Liquid particulates dispersed in air are called "mists" or "fogs," and the term "aerosol" is used collectively for air suspensions of liquid and solid particulates.

Often an atmosphere being sampled contains a combination of contaminants. Caution must be exercised when samples are taken and when the data from atmospheres of this nature is interpreted.

NATURALLY-OCCURRING AIRBORNE RADIOACTIVITY

Man's environment has always included airborne radioactivity, primarily because of the uranium and thorium content of the soil. The uranium decay series and the thorium decay series each produces an isotope of radon, which, not being chemically reactive, can migrate to the air. Figure 20-3-1 gives the uranium and thorium decay series and indicates the possible airborne decay products from the respective isotopes of radon. ^{220}Rn and ^{222}Rn, along with their radioactive daughters, comprise the major portion of naturally-occurring airborne radioactivity.

There is a rather wide variation in this natural activity, based on time and geography. Geographical variation occurs because the uranium and thorium content of all soils is not uniform and because all soils do not liberate radon readily. Moisture content seems to affect these factors considerably. Air masses that move from over large bodies of water have been shown to have low concentrations of these isotopes.

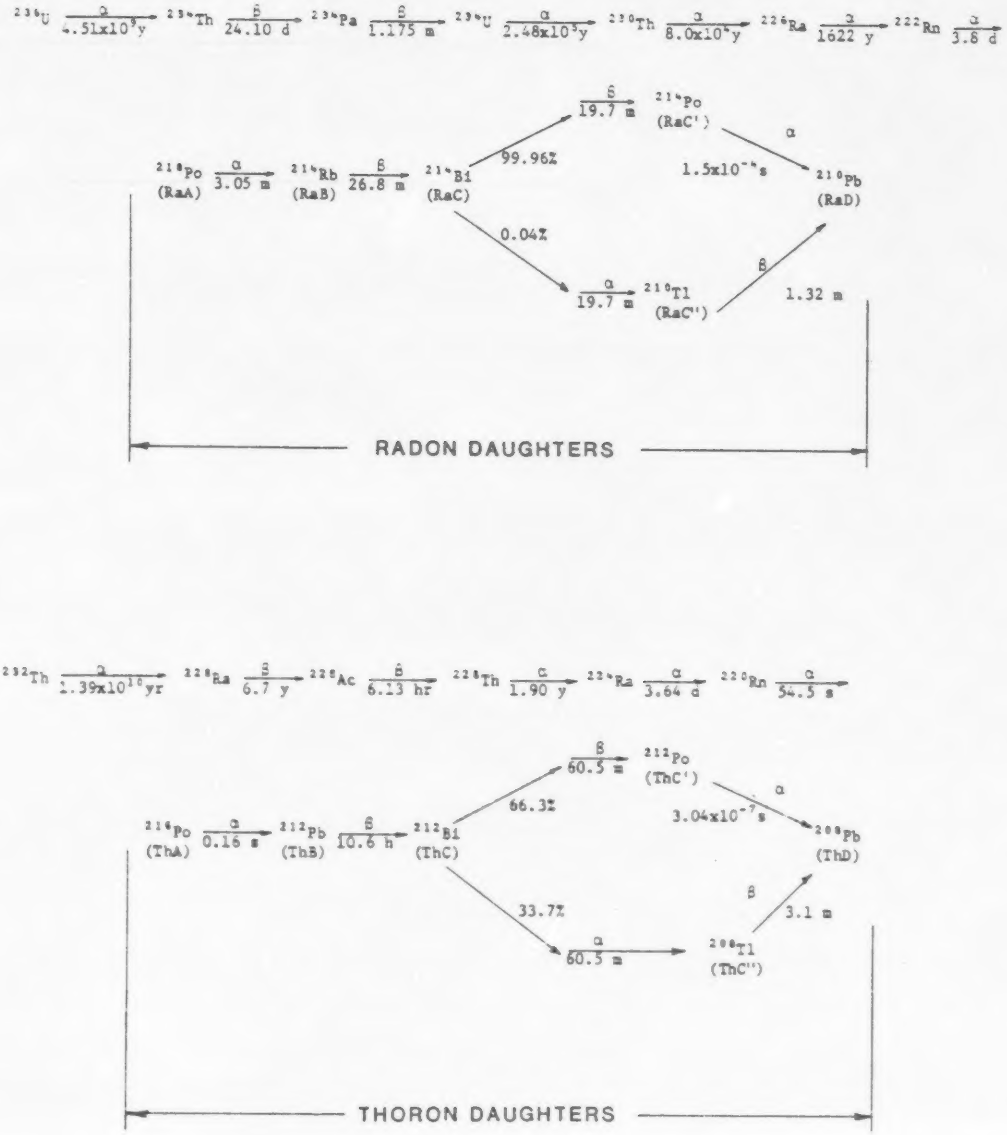

²³⁸U α 4.51x10⁹y ²³⁴Th β 24.10 d ²³⁴Pa β 1.175 m ²³⁴U α 2.48x10⁵y ²³⁰Th α 8.0x10⁴y ²²⁶Ra α 1622 y ²²²Rn α 3.8 d
²¹⁸Po (RaA) α 3.05 m
²¹⁴Rb (RaB) β 26.8 m
²¹⁴Bi (RaC)
99.96%
β 19.7 m
²¹⁴Po (RaC')
α
1.5x10⁻⁴s
²¹⁰Pb (RaD)
0.04%
α 19.7 m
²¹⁰Tl (RaC'')
β
1.32 m
RADON DAUGHTERS
²³²Th α 1.39x10¹⁰yr ²²⁸Ra β 6.7 y ²²⁸Ac β 6.13 hr ²²⁸Th α 1.90 y ²²⁴Ra α 3.64 d ²²⁰Rn α 54.5 s
²¹⁶Po (ThA) α 0.16 s
²¹²Pb (ThB) β 10.6 h
²¹²Bi (ThC)
66.3%
β 60.5 m
²¹²Po (ThC')
α
3.04x10⁻⁷s
²⁰⁸Pb (ThD)
33.7%
α 60.5 m
²⁰⁸Tl (ThC'')
β
3.1 m
THORON DAUGHTERS

Figure 20-3-1. Uranium and thorium decay series.

At any given location, natural airborne radioactivity varies with changes in temperature, humidity, wind speed, and wind direction. Typically, concentration is the greatest in the morning, decreasing in the afternoon and beginning to increase again in the early morning. The early morning increase is attributed to an increase in surface atmospheric stability and to a decrease in wind velocity. The afternoon decrease in concentration is caused by vertical transport by convection (transference of heat by movement of the atmosphere) due to solar heating of the surface of the earth. Variations occur both seasonally and diurnally.

The radon/thoron ratio is also variable, with values that have ranged from about 1 to 6, with a six-month average value of 4.7. Although thorium is more abundant than uranium, ^{222}Rn (radon) with a half-life of 3.8 days has a greater probability of migrating to the air than ^{220}Rn (thoron) which has a half-life of only 54.5 seconds.

Decay products of radon and thoron are electrostatically charged particulates that readily attach themselves to any dust in the air. Usually, they are associated with the smallest atmospheric dust particles, which range in size from 0.005 μ to 0.04 μ. These radioactive particles, when captured on filters, may be of such high activity that they completely mask the activity of the isotope of interest, particularly if the filter is counted shortly after the sample is collected.

This activity varies so unpredictably that it is a great hindrance to interpretation of air sample data. Some schemes for relieving this problem will be discussed in the section on air filter sample analysis.

REPRESENTATIVE SAMPLING

There are two related criteria to be considered in collection of a representative sample. The first of these is sample concentration; that is, concentration of the materials in the air entering the sampler must be the same as that near the orifice of the sampler. This is particularly true in the sampling of a stack for particulates. In this case, care must be taken to ensure that the sampling is "isokinetic," or that the linear air flow rate into the sampling orifice is the same as air velocity around the orifice. Sampling that is not isokinetic will discriminate against large or small particles, depending upon conditions.

The second criterion is that the concentration in the air at the sampling head must be representative of the concentration at the point of concern. Considerable variation has been reported between samples collected by a personal air sampler and a fixed sampler located in the same room. Therefore, conditions of the sampling area must be observed when a sampling program is designed for specific areas.

SAMPLING METHODS AND DEVICES

The two fundamental types of samples are –

- An integrated sample, in which the contaminants are concentrated in a collecting medium. This type of sample represents an average of the collection time and will not reveal variations in the air concentration.
- A grab sample, in which a part of the air is isolated with the contaminant in its original concentration.

This type of sample will indicate conditions at a point in time and space. However, many samples of this type are required before an attempt can be made to describe the conditions over a large area.

Either type of sample may be taken for either gases or particulates.

Choice of sample type, method, and device is directly involved with the choice of analytical method. Additional concerns are characteristic of the contaminant and its allowable levels in the atmosphere. In general, a less sensitive method of analysis requires a larger volume of air to be sampled. When sampling for radioactivity, the most prominent characteristic of the radioisotope (that is, its radioactivity) usually is the basis for any analytical procedure to be used.

Choice of sampler type is dictated by the type of information required. When immediate information is needed, collection and analysis are performed at the same time. If immediate information is unnecessary, the analysis usually is done at a laboratory removed from the sampling site. This allows use of less bulky and somewhat more economical samplers, and the analysis is more flexible and frequently better.

FILTER SAMPLERS

Sampling by filtration is the most common method of air sampling for radioactive particulates. It is both simple and efficient.

A wide choice of filters is available, with a type available for almost any specific purpose. Characteristics that should influence filter choice are collection efficiency, flow resistance, and mechanical strength. Because no one filter is best at all of these factors, a compromise is necessary.

The sampling technique is to draw air at a known flow rate through a filter for a known length of time. Figure 20-3-2 represents a typical filter system.

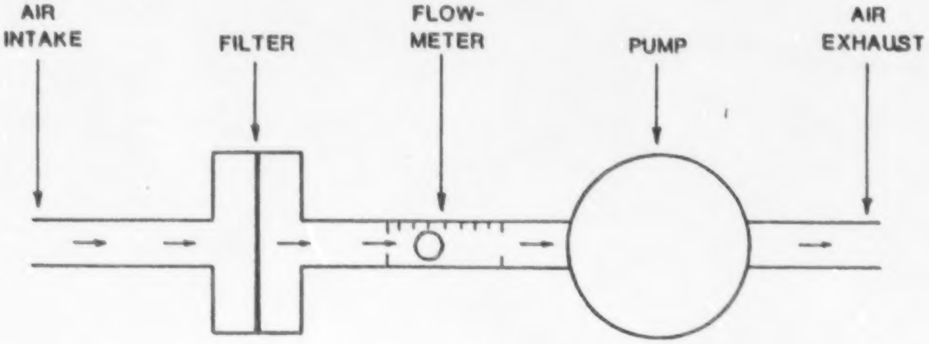


Figure 20-3-2. A filter sampler system.

Three different types of filter samplers are in common use throughout the nuclear industry: the low-volume sampler, the high-volume sampler, and the continuous sampler.

Low-volume samplers normally consist of some type of blower/vacuum motor, with an air flow meter mounted between the filter and vacuum intake. Air flow rates may vary from 15 to 35 liters per minute. Figure 20-3-3 is an example of a low-volume sampler.

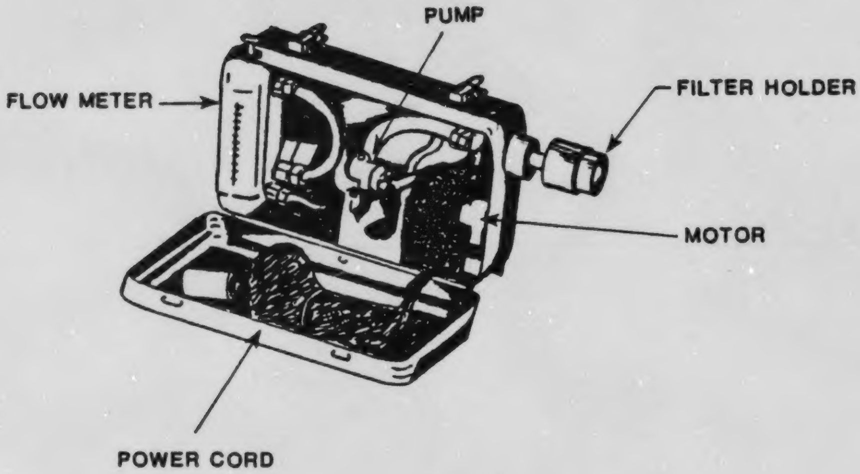


Figure 20-3-3. A low-volume air sampler.

High-volume samplers commonly are utilized for short collection times. These units may sample at rates of up to 650 liters per minute, and are similar in construction to the low-volume samplers, except that they have much larger vacuum units and much more powerful motors. A high-volume sampler is illustrated in Figure 20-3-4.

Continuous samplers almost always employ a positive-displacement vacuum pump or blower; and are utilized when samples are to be taken continuously over several days. These units must be checked periodically when in operation to see that proper lubrication is maintained. Air flow rates may be as high as 60 liters per minute. Figure 20-3-5 displays an example of a continuous sampler.

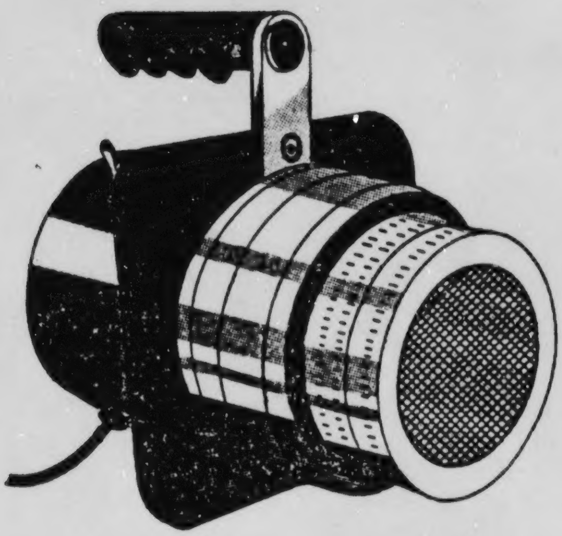


Figure 20-3-4. A high-volume air sampler.

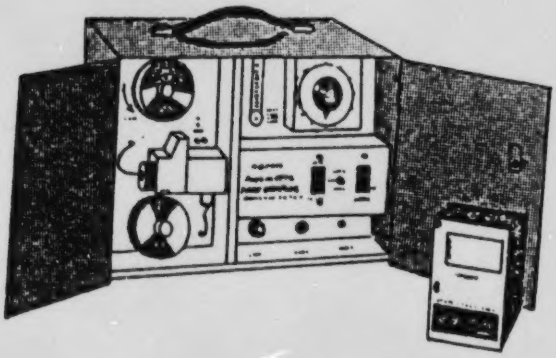


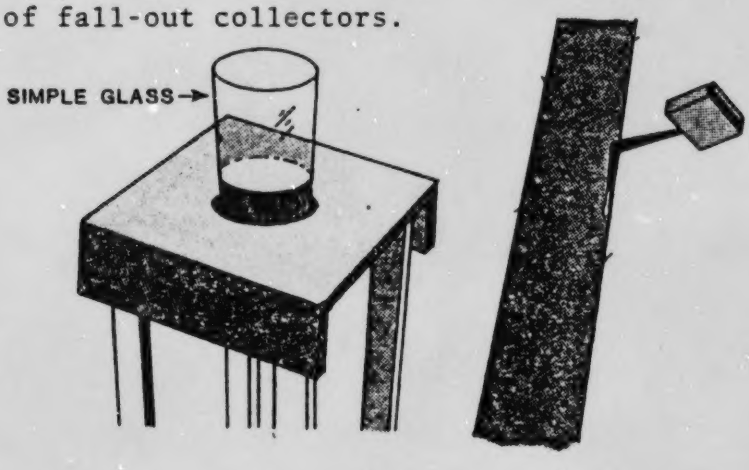
Figure 20-3-5. A continuous air sampler.

SEDIMENTATION SAMPLERS

A sedimentation sampler is a device that collects particulates that settle into it. This method of sampling is important for particulates or aerosols only when the particles are larger than 10 micra. Smaller particles settle so slowly that convection currents prevent sustained downward motion.

Dust-fall jars either may be used dry or with a collecting liquid, usually water. When a wet sampler is utilized during summer months, an algal suppressant may have to be added to the water. The suppressant in most general use is quaternary ammonium chloride and copper sulfate. During winter months, a volatile antifreeze, such as isopropanol 1, may be required to prevent freezing.

Wet samples are evaporated to dryness before being counted, and the dry sample is weighed. After counting, the results are reported as μCi/mg/cm^2/unit time, i.e., 5 x 10^{-4} μCi/mg/cm^2/day. Figure 20-3-6 illustrates two types of fall-out collectors.



(a)
FALL-OUT JAR

(b)
FALL-OUT TRAY

Figure 20-3-6. Fall-out collectors.

ELECTROSTATIC PRECIPITATORS

Electrostatic precipitation is one of the most efficient methods of particulate collection. The method imposes an electric field at right angles to the air flow, ionizes the particles, and collects them on a charged electrode.

The collector is usually a glass or metal cylinder mounted concentric to a high-voltage (10–20 kV) electrode. The collector may be removed after operation and counted directly, or the active precipitate may be washed from the collector for counting.

These collectors are very efficient, and proper attention to the parameters of potential, air flow, and collector size yields consistently reproducible efficiencies approaching 100%. Figure 20-3-7 depicts one of the most popular electrostatic precipitators.

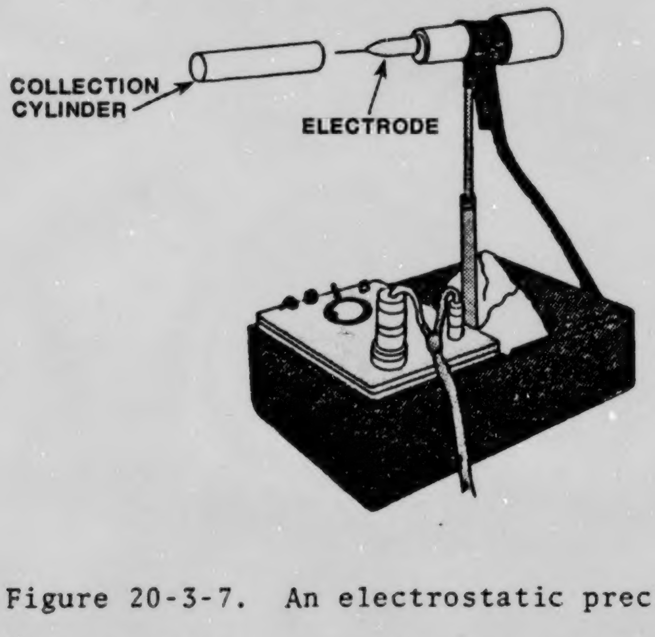


Figure 20-3-7. An electrostatic precipitator.

GRAB SAMPLES

A partially evacuated container of known volume can be used to sample for airborne gases or particulates. The container usually is connected to a vacuum pump, evacuated, isolated, and then removed to the sampling location. There it is opened to admit the atmosphere to be sampled, then isolated, and removed for analysis. However, the collecting vessel may not be evacuated prior to collection. The vessel may be opened and the sample collected by purging of the collection vessel. The vessel is purged by suctioning the atmosphere to be sampled through the vessel. This procedure requires suctioning of at least 10 times the volume of the container volume through the container.

Grab samples are relatively inefficient for sampling particulates, primarily because there is no magnification factor to be obtained. However, there are times when grab samples are the only method to be employed. Figure 20-3-8 is a diagram of one type of grab sampler.

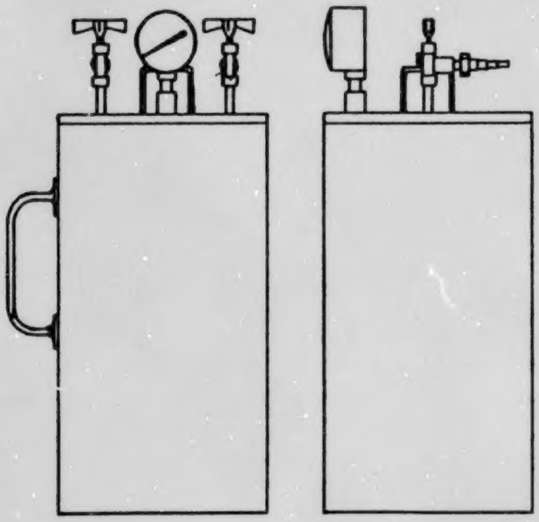


Figure 20-3-8. A grab sampler.

IMPINGERS

Impingers remove particles from an air stream by inertial precipitation. The air stream impinges against a surface at right angles, and then continues. Particles above a certain size cannot follow after the impingement (because of their inertia) and are collected.

The impingement surface often is greased to enhance collection of particulates, or the surface may be immersed in a liquid that serves as a collecting medium. In the latter case, an aliquot of the liquid may be counted.

The Greenburg-Smith impinger and its smaller look-alike, the midget impinger, are instruments of the latter type. Sampling rates for these instruments are approximately 9 to 0.9 liters per minute, respectively; and the lower limit of collection is approximately 0.7 μ. Figure 20-3-9 illustrates the Greenburg-Smith and midget impingers.

Another type of impinger is the cascade impactor. This type of sampler consists of a series of four impingement slots, each above a separate collection plate. The instrument is arranged in such a manner that the slots become progressively smaller, thereby progressively increasing velocity of air through the jets. Additionally, the distance between the slot and the plate becomes progressively smaller. This structure permits classification of particulates according to size, the largest being collected on the first slide and the smallest collected on the last side. Figure 20-3-10 is a representation of a cascade impactor.

Still another version of the impinger is the Venturi scrubber. Venturi scrubbers usually employ water as the collecting medium. Water is induced into the throat of the

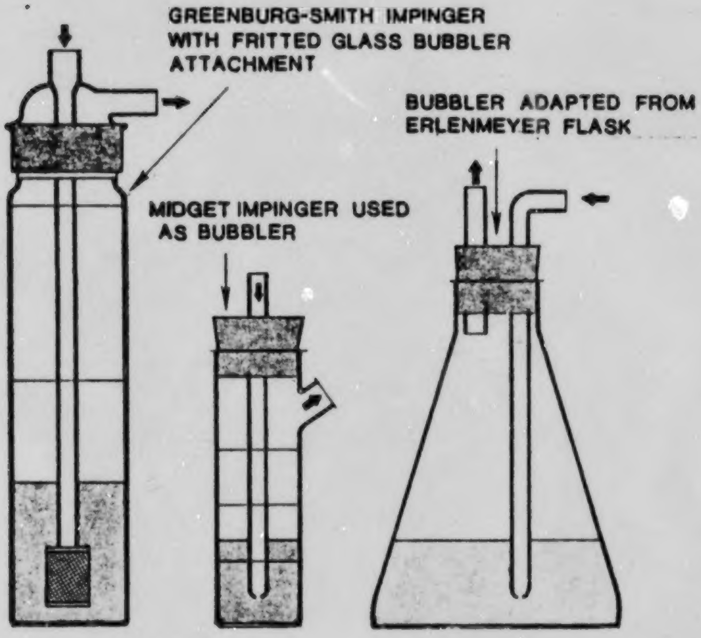

GREENBURG-SMITH IMPINGER WITH FRITTED GLASS BUBBLER ATTACHMENT
BUBBLER ADAPTED FROM ERLENMEYER FLASK
MIDGET IMPINGER USED AS BUBBLER

Figure 20-3-9. The Greenburg-Smith and midget impingers.

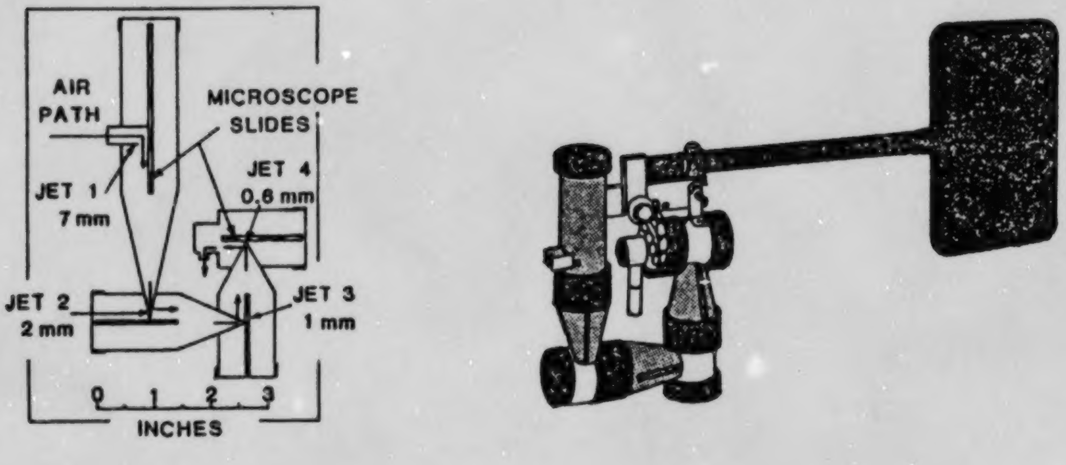

AIR PATH
MICROSCOPE SLIDES
JET 1 7 mm
JET 4 0.6 mm
JET 2 2 mm
JET 3 1 mm
0
1
2
3
INCHES

Figure 20-3-10. A cascade impactor.

Venturi tube and is atomized into small droplets. The smallness of the water droplets and the high relative velocities between these water droplets and the particles provide excellent impingement conditions. The particles are collected effectively by impingement upon the droplets and are retained in the apparatus. Figure 20-3-11 is a diagram of a Venturi scrubber.

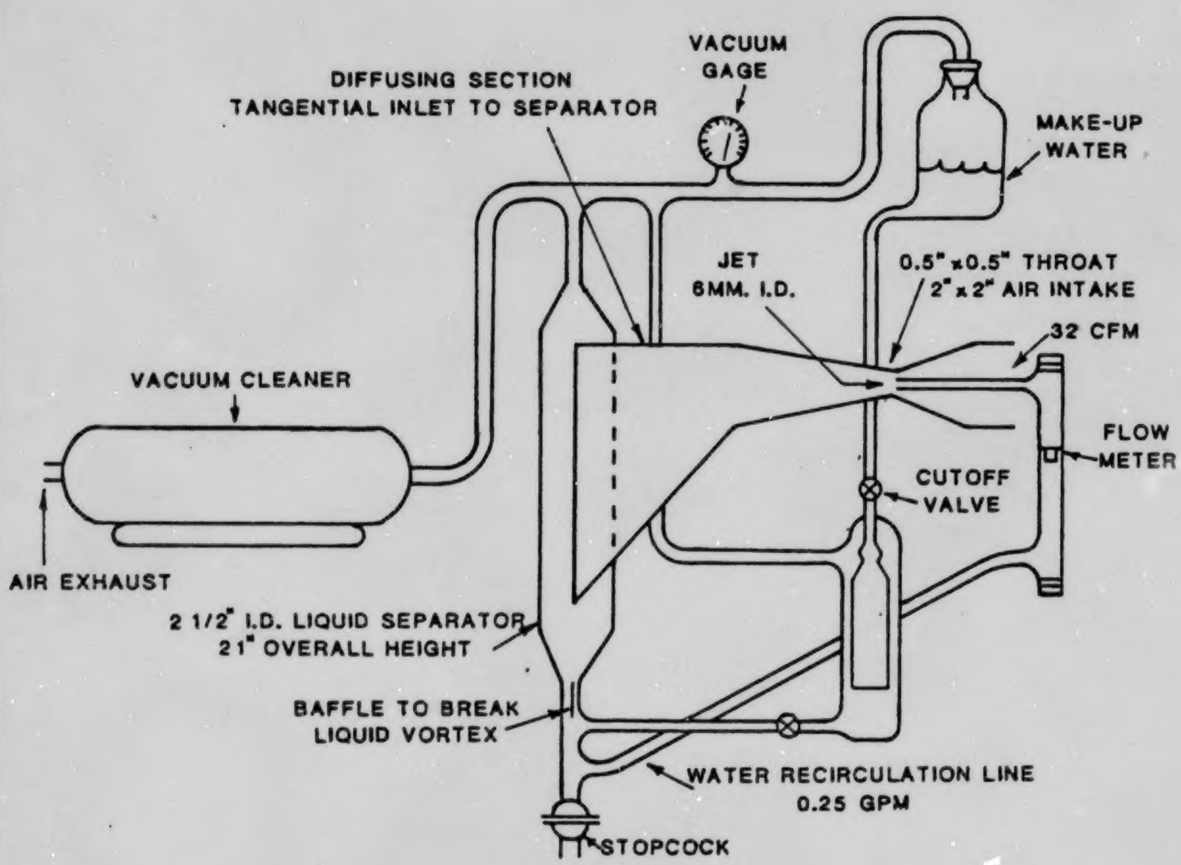

VACUUM GAGE
DIFFUSING SECTION
TANGENTIAL INLET TO SEPARATOR
MAKE-UP WATER
JET
6MM. I.D.
0.5" x 0.5" THROAT
2" x 2" AIR INTAKE
32 CFM
VACUUM CLEANER
FLOW METER
CUTOFF VALVE
AIR EXHAUST
2 1/2" I.D. LIQUID SEPARATOR
21" OVERALL HEIGHT
BAFFLE TO BREAK
LIQUID VORTEX
WATER RECIRCULATION LINE
0.25 GPM
STOPCOCK

Figure 20-3-11. Diagram of a Venturi scrubber.

In addition to radioactive analysis, particulate samples collected by any of the inertial devices may be weighed, analyzed for specific contaminants, or examined microscopically. Results may be reported in μCi/unit volume of air sampled, μCi/mr/unit volume of air sampled, or in μCi/number of particles/unit volume sampled.

FILTERS

Sieve action is not the only mechanism at work in the filtering process. Not only are particles larger than the filter pore size retained, but there is also highly efficient retention of particles smaller than pore size. Relatively large particles are impinged on the filter fibers because of airstream deflection. Increase in the airstream velocity effectively decreases this method of filtration.

Other large particles experience frictional force, behaving in a manner outlined in Stokes' Law, and settle onto the filter fibers. Additional particles may be intercepted directly by the filter fibers. Collection and/or retention on some filters is aided by electrostatic forces. Likewise, humidity may be directly involved. Brownian motion (random motion of small suspended particles) plays a part in bringing very small particles onto the filters.

Usually, there will be an optimum airflow for a given particulate.

CELLULOSIC FIBROUS PAPERS

The most common cellulosic fibrous paper filters are the Whatman types produced for analytical chemistry. Whatman Nos. 32, 42, 44, and 50 have a high removal efficiency. However, they also have a high resistance to air flow.

Efficiency of most filters, regardless of type, improves during sampling, due to buildup of an additional filter mat of the material being collected. The buildup presents a problem with filter papers: They have a tendency to plug up and to decrease the flow rate. For this reason, filter papers usually are not acceptable when large quantities of particulates are to be collected at high flow rates.

The use of cellulose filters is not recommended for collection of samples to be examined for alpha activity. Particulates have a tendency to penetrate deeply into the filter material – greatly affecting the efficiency of alpha radiation detection.

CONTINUOUS SAMPLERS

Another method of air particulate monitoring involves use of a continuous air sampler (Figure 20-3-12). A continuous air sampler contains a roll of filter paper, continuously advanced and continuously counted. This device usually is equipped with a recorder and an alarm system. It may have provisions for alpha counting, beta counting, beta + gamma counting, gamma counting, or gamma counting of a photon of specific energy. Systems may be designed with any combination to accomplish a specific job or jobs.

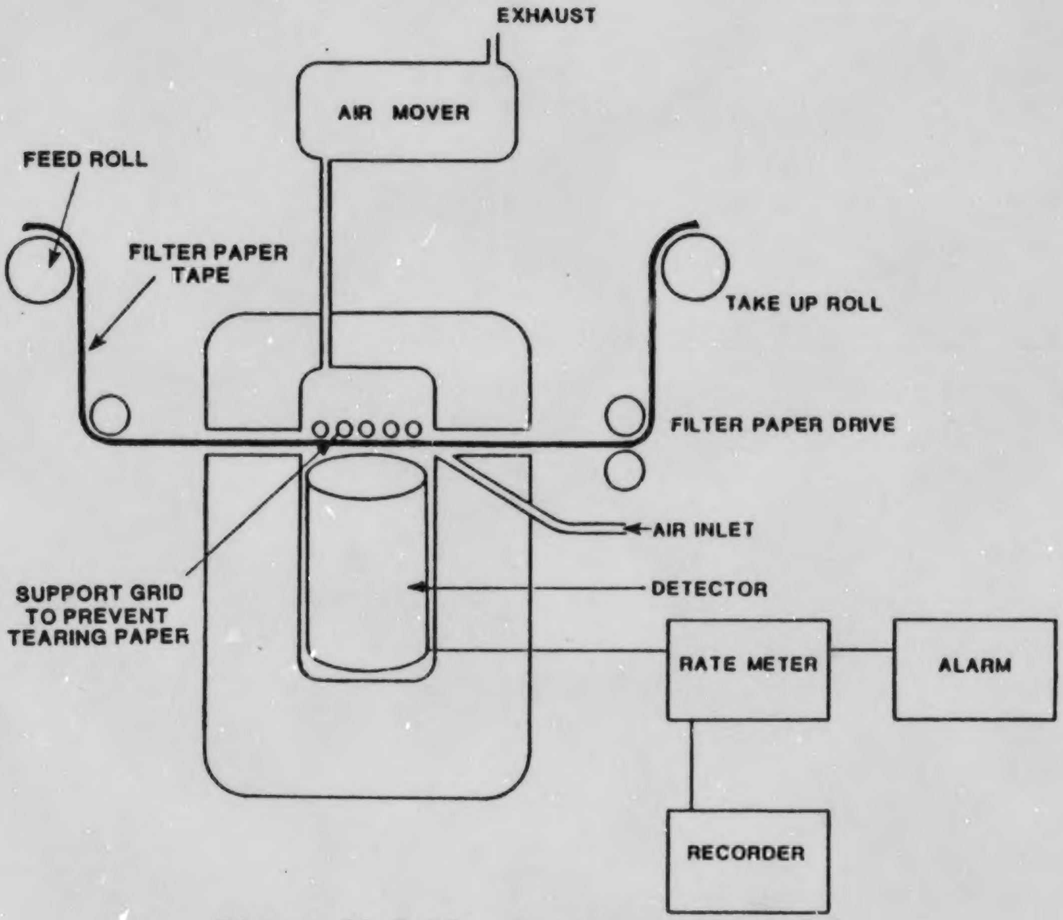

EXHAUST
AIR MOVER
FEED ROLL
FILTER PAPER TAPE
TAKE UP ROLL
FILTER PAPER DRIVE
AIR INLET
DETECTOR
SUPPORT GRID TO PREVENT TEARING PAPER
RATE METER
ALARM
RECORDER

Figure 20-3-12. Representation of a continuous air sampler.

MEMBRANE FILTERS

Membrane filters are made of thin films of material, usually a cellulose ester, with conically shaped pores. The peak of the cone forms the filtering surface. Pore sizes range from 0.2 micra to 7 micra, depending upon the type of filter.

Two features cause the membrane filter to be very useful in analysis of radioactive materials. First, due to electrostatic attraction, these filters are capable of retaining particles much smaller than their average pore diameter. Second, the particles are collected on, or very close to, the filter surface. This is especially desirable in working with alpha emitters or weak beta emitters.

There are also two major disadvantages. Most membrane filters are attacked by organic solvents, severely limiting their use in the sampling of organic materials. Additionally, there is a very high pressure drop across the filter at high flow rates. There are methods with which the drop can be compensated, but they are expensive.

GLASS FIBER FILTERS

Glass fiber filters provide good strength, high efficiency, and chemical resistance. They are produced from a finely spun glass fiber, which is formed into the filter mat and then flash-fired to remove any organics.

These filters provide an efficiency of practically 100%, and both beta and gamma activities are easily measured. However, they are not suitable for alpha activity.

FILTER SAMPLE ANALYSIS

A filter sample collected from air that contains a single radioactive nuclide has a continued increase in activity during the time of sampling. The buildup of radioactivity stops as soon as collection of radioactive particulates ceases. When collection ceases, sample activity begins to decrease because of radioactive decay. Figure 20-3-13 graphically presents buildup of activity on a filter and its decay after sampling ends.

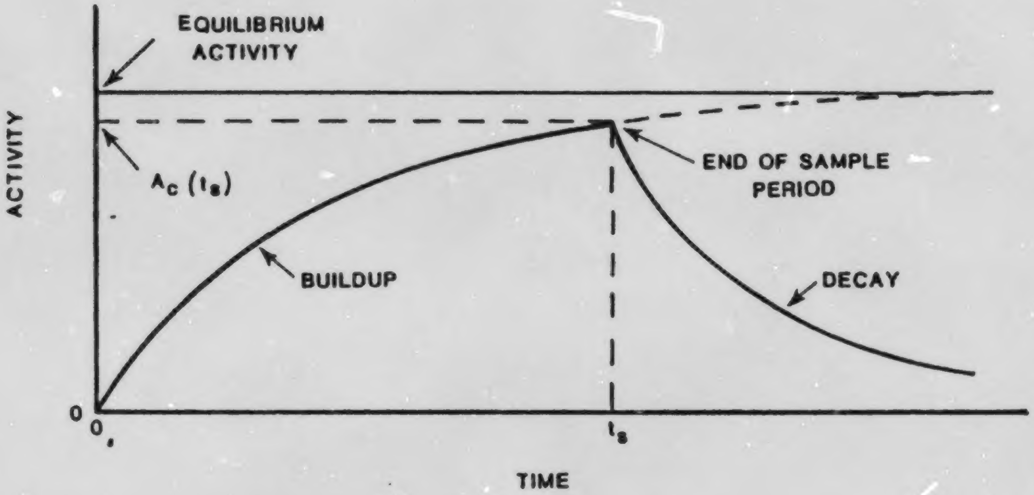


Figure 20-3-13. Activity of a single radionuclide on a filter.

Regrettably, sampling seldom occurs under such ideal conditions. The naturally-occurring airborne radioactive materials usually are neither zero nor predictable. Therefore, the total activity on a filter at any time is the sum of the activities of radon, thoron, and their daughters, and of the radioactive material of interest.

The problem is to determine activity of the nuclide of interest in order that its concentration can be calculated. The activity of interest can be determined if the isotope is of sufficiently longer half-life than the daughters of thoron. Determination requires several hours, by which time the information may have become moot.

A first assessment of air contamination may be made by assuming that all activity on the paper is due to the isotope of interest and that no decay has occurred. (The assumption is made that the half-life is considerably longer than the sampling time.) This procedure is acceptable for use with beta emitters, but will not work with alpha emitters. Variation in the concentration of naturally-occurring airborne alpha emitters is so great that maximum permissible concentration (MPC) values of other isotopes may be obscured completely.

FIRST-COUNT FACTOR

A somewhat more reliable early determination of an airborne radioactive particulate hazard can be achieved by finding the ratio of the activities of beta emitters to alpha emitters on the filter. This determination must be made as soon as possible after the sample is collected. Equation 20-3-1 is utilized for the calculation of the "first-count factor."

$$\Gamma = \frac{(\beta\gamma)\ \text{dpm/m}^3}{(\alpha)\ \text{dpm/m}^3}$$

Equation 20-3-1
(Equation = Eq.)

where:

Γ = first-count factor.

Actual numbers of the ratio will vary with the counting system used, with efficiencies, etc. Therefore, an average value for Γ must be determined from an air sampling history developed for a clean area. Usually, the average value will vary through a range from 1.1 to 2.7. When this average value has been determined, an increase in the ratio will indicate unnatural beta contamination, and a decrease in the ratio will indicate presence of unnatural alpha contamination. NOTE: This method should not be used to estimate allowable concentrations, but to detect an alpha or beta air contamination.

CONCENTRATION OF LONG-LIVED ISOTOPES

The daughters of radon have a composite half-life of approximately 35 minutes. Therefore, if the filter sample is not counted for about 6 hours after collection, the radon daughter activity will be reduced by a factor of 10^3 and can be ignored. Activity at this time is due to activity of the isotope of interest and activity of the thoron daughters.

Daughters of thoron have a composite half-life of 10.64 hours. If half-life of the isotope of interest is significantly longer than the thoron daughter composite, activity of the isotope of interest can be determined.

The general practice is to count the sample 6 hours after collection and again 16 hours after collection, or at other times, ensuring that there is approximately 10 hours between counts and that the first count is taken not less than 6 hours after collection. The thoron daughter activity ratio can be predicted at different counting times, and the fraction of the activity due to the contaminant can be calcuated according to Equation 20-3-2.

$$A_c(t_2) = A_2 \frac{e^{\lambda(Th)^{\Delta t}} - \frac{A_1}{A_2}}{e^{\lambda(Th)^{\Delta t}} - 1} \qquad \text{Eq. 20-3-2}$$

However, in practice, the value usually is taken from a nomogram such as that in Figure 20-3-14, after the alpha counting ratios and the beta counting ratios are counted and calculated according to Equations 20-3-3 and 20-3-4.

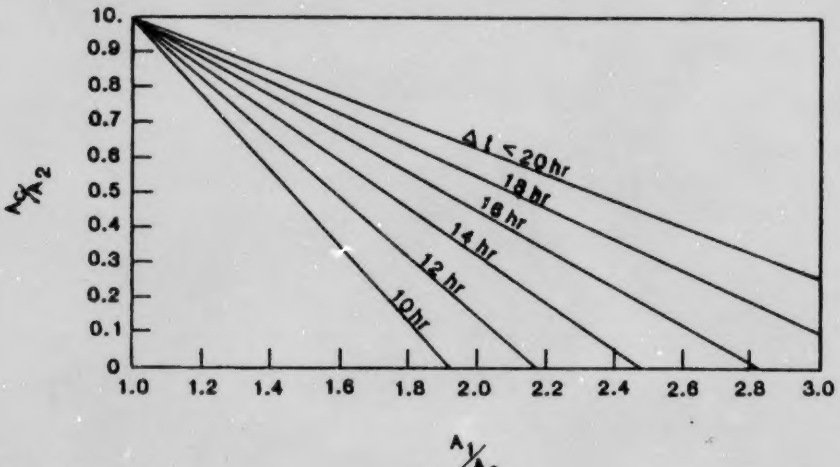


Figure 20-3-14. Contaminant activity fraction-versus-activity ratio.

$$A_\alpha = \frac{A_\alpha(t_1)}{A_\alpha(t_2)} \qquad \text{Eq. 20-3-3}$$

$$A_\beta = \frac{A_\beta(t_1)}{A_\beta(t_2)} \qquad \text{Eq. 20-3-4}$$

The first count ratio may or may not give an indication of the presence of a contaminant.

After the second counting period (i.e., 10 hours after the first count period), actual activity caused by the contaminant can be calculated.

EXAMPLE

Given: Shortly after a suspected release of ^{239}Pu, a high-volume air sample is taken for 60 minutes at an average flow rate of 2 m^3 per minute. The following disintegration rates are obtained from the filter:

Activity of filter	disintegrations per minute		
	at time of sample	after 6 hr	after 16 hr
A_β	15,000	1,500	780
A_α	8,100	1,800	1,500

Find: (a) Suspicion of contamination based on first count ratio.

(b) Activity and concentration of contamination based on counts 6 and 16 hours after sampling.

Solution: (a) Calculate the first count ratio from Equation 20-3-1.

$$\Gamma = \frac{(\beta\gamma)\ \text{dpm/m}^3}{(\alpha)\ \text{dpm/m}^3}$$

$$= \frac{15{,}000\ \text{dpm}/120\ \text{m}^3}{8{,}100\ \text{dpm}/120\ \text{m}^3}$$

$$\Gamma = 1.85$$

113

Because the first count ratio falls within the normal variation range of 1.7 to 2.7, there can be no confirmation or rejection of the suspected ^{239}Pu contamination at this time.

(b) Determine activity and concentration of the contamination based on counts 6 and 16 hours after sampling.

- Calculate from Equations 20-3-3 and 20-3-4 the activity ratios for both alpha and beta activities using disintegration rates at 6 and 16 hours after sampling.

$$A_\alpha = \frac{A_\alpha(t_1)}{A_\alpha(t_2)}$$

$$= \frac{1{,}800 \text{ dpm}}{1{,}500 \text{ dpm}}$$

$$A_\alpha = 1.2.$$

$$A_\beta = \frac{A_\beta(t_1)}{A_\beta(t_2)}$$

$$= \frac{1{,}500 \text{ dpm}}{780 \text{ dpm}}$$

$$A_\beta = 1.92.$$

- Use calculated alpha ratio and beta ratio to determine the portion of the activity due to contamination after 16 hours. Refer to the nomogram in Figure 20-3-14. The portion of the alpha activity caused by contamination is determined by locating the activity ratio on the ordinate of the nomogram, drawing a line parallel to the abcissa until it intersects with the line corresponding to the sampling time, and reading the value of the activity portion in the abcissa at a point opposite the point of intersection. Because the calculated activity ratio for the alpha activity is 1.2 and the time between counts was 10 hrs, the alpha contaminant activity fraction from the nomogram is 0.78. By use of the same nomogram and the beta activity ratio of 1.92 and Δt of 10 hours, the beta contaminant activity fraction is 0.00. Therefore, all the contamination activity is due to alpha activity.

- Calculate the amount of contamination due to ^{239}Pu.

$$A_{Pu} = \text{Contaminant activity fraction x activity of filter}$$
$$= 0.78 \times 1{,}500 \text{ dpm}$$
$$A_{Pu} = 1170 \text{ dpm.}$$

- Calculate concentration of ^{239}Pu.

$$C_{Pu} = \frac{A_{Pu}}{\text{Volume of Sample}}$$

$$C_{Pu} = \frac{1170 \text{ dpm}}{120 \text{ m}^3}$$

$$C_{Pu} = 9.75 \text{ dpm/m}^3.$$

This procedure greatly simplifies calculation of activity caused by contamination. Without the procedure, the technician would be forced to wait until six days after the sampling, when radon, thoron, and their daughters have decayed out. At that point, any measured activity would be due to contamination, assuming that the contaminant was very long lived.

SHORT-LIVED EMITTERS

When the half-life of a contaminant begins to approach that of the daughters of thoron (i.e., 10.6 hours), calculation of the concentration becomes considerably more difficult with the procedures discussed so far. All of these radioisotopes of short half-life, however, have MPC values sufficiently large so that any activity-concentration that would be of interest literally would cover completely the radon-thoron daughter activities on the filter. The radon-thoron daughter activities then can be ignored and the activities of the contaminants can be calculated directly from the alpha and beta-gamma activities. Consideration must be given to decay during and after sampling, in order to prevent under-estimation of activities.

Additionally, during sampling for very short half-lived radioisotopes (i.e., half-life less than 35 minutes), a high-volume sampler should be used, and the sampling time should not be longer than 3 half-lives of the isotope sought. Longer sampling simply would cover part of the activity with radon and its daughters.

FILTER SAMPLE PREPARATION

Samples collected on filters require no preliminary treatment if they are to be analyzed by the preceding methods. Subsequent analyses may be necessary, however, for specific elements. These may be performed only if the samples are subjected to special treatment before radiochemical analysis is begun. This is done after gross-alpha and gross-beta counting 6 days after collection.

GLASS FIBER FILTER SAMPLES

This preparation requires that a measured portion of the filter be fused with sodium carbonate and the melt dissolved. The silica present is destroyed by treatment with hydrofluoric acid, and the remaining salts are dissolved and diluted to a known volume with hydrochloric acid. The stepwise procedure is thus:

1. Weigh a portion of the filter; place in a platinum crucible.
2. Cover sample with 3 times its weight of sodium carbonate.
3. Place crucible and sample in a muffel furnace; fuse the sample at 950 ± 20°C for 30 minutes.
4. Allow crucible to cool.

5. Leach residue in the crucible by adding 50 ml of distilled water and heating the crucible on a hot plate until the liquid just begins to boil.
6. Transfer liquid and solid to a beaker.
7. Neutralize the solution with concentrated HCl.
8. Estimate the volume of the solution; add sufficient HCl to make the solution 2N in HCl.
9. Filter the residue, using Whatman No. 40 filter paper, collecting and saving the filtrate. Wash residue twice with 5-ml portions of 2N HCl and twice with 5-ml portions of distilled water. Add washings to the filtrate.
10. Transfer filter paper to a platinum crucible; ignite in a furnace.
11. Cool crucible; add 10 ml of concentrated HF; evaporate to dryness on a hot plate.
12. Add 5 ml of 6N HCl; evaporate to dryness on a hot plate.
13. Repeat Step 12.
14. Dissolve the chloride salts in the crucible with 2N HCl; combine with the filtrate in Step 9.
15. Dilute solution to a known volume with 2 N HCl. This solution then may be used for the analysis for Pu, U, Sr, Y, Ce, Ba, Cs, Zr.

CELLULOSE FIBER FILTER SAMPLES

The treatment of cellulose fiber filters is somewhat simpler than that for glass fiber filters. The cellulose filter paper is ignited; the ash is treated with hydrofluoric acid to remove silica; and the remaining chlorides are dissolved in dilute hydrochloric acid. The stepwise procedure is thus:

1. Place filter sample in a platinum crucible; ignite in a muffel furnace.
2. Cool crucible; add 10 ml of concentrated hydrofluoric acid.
3. Evaporate to dryness on a hot plate.
4. Add 5 ml of 6N HCl; evaporate to dryness on a hot plate.
5. Repeat Step 4.
6. Dissolve the remaining chlorides in 2N HCl, quantitatively transfer to a volumetric flask, and dilute with 2N HCl. The solution then is ready for analysis.

MEMBRANE FILTER SAMPLES

Membrane filters are dissolved with acetone, evaporated to dryness, and ignited. The ash then is treated with HF to remove silica, and the remaining salts are dissolved in dilute hydrochloric acid. The stepwise procedure is thus:

1. Place membrane filter sample in a platinum crucible; add 5 ml of acetone to dissolve the membrane.
2. After membrane is dissolved, evaporate solution to dryness.
3. Ignite residue in a muffel furnace.
4. Cool crucible; add 10 ml of concentrated HF.
5. Evaporate to dryness on a hot plate.
6. Add 5 ml of 6N HCl; evaporate to dryness on a hot plate.
7. Repeat Step 6.

8. Dissolve remaining chlorides in 2N HCl. Quantitatively transfer to a volumetric flask, and dilute with 2 N HCl. The solution then is ready for analysis.

RADIOCHEMICAL ANALYSIS

The following procedures have been found to be effective in analyzing air particulate samples collected on filters. Solutions from the preparatory procedures already discussed are suitable for analysis.

PLUTONIUM

Plutonium content from air samples collected on filter papers is determined by electrodeposition. The soluble chloride of plutonium is reduced to the trivalent state and coprecipitated as the fluoride with lanthanum as a carrier. The fluoride precipitate then is dissolved in an acid aluminum nitrate solution, and the plutonium is extracted into thenyltrifluoroacetone (TTA). The plutonium then is back extracted into strong acid, oxidized, and electrodeposited on stainless steel for alpha counting. The stepwise procedure will be given in the procedures section of this module.

URANIUM

Uranium content of air samples, where the uranium has been treated to convert it to the soluble chloride form, is determined by separating it from radionuclides that would interfere (i.e., potassium-40, throium, and radium). The uranium then is electroplated on a planchet for alpha counting.

The method of separation is quite direct. The soluble uranium, as the chloride, is adsorbed onto a strongly basic ion-exchange column that has been pretreated with 6N HCl. All interfering ions are eluted with 9N HCl. The uranium then is eluted by the passing of 1N HCl through the column. The iron usually found with uranium also will be eluted, but it will not interfere with the analysis. After elution, the uranium is converted to the nitrate, dissolved in dilute ammonium oxalate, and electrodeposited on a planchet for alpha counting. The stepwise procedure for this preparation will be given in the procedures section of this module.

STRONTIUM

Humans absorb very little radiostrontium by inhalation. However, analysis of the atmospheric content of radiostrontium is a very good indicator of other fission products.

Strontium and barium carriers are added to the acid chloride solution of the sample. The alkaline earths then are precipitated as the carbonates, then converted to the nitrates, precipitating barium and strontium as the nitrate. Barium and strontium nitrates are dissolved in distilled water and purified by scavenging with hydroxides. Barium and radium are separated from the solution by chromate precipitation. The solution is acidified, yttrium carrier is added, and the solution is stored to allow growth of ^{90}Y activity.

Yttrium then is precipitated from the solution as the hydroxide. The hydroxide precipitate is dissolved in HCl, and yttrium oxalate is precipitated, dried, weighed, and beta counted.

Strontium then is precipitated as the carbonate, dried, weighed, and beta counted to determine the total strontium activity. Strontium-89 activity is calculated as the difference between the total strontium activity and the activity due to ^{90}Sr, counted as ^{90}Y. A stepwise procedure for this analysis is given in the procedures section of this module.

EXERCISES

1. Given a filter sample that has a beta activity of 29,800 dpm at the time of collection, an activity of 18,200 dpm 6 hours after collection and 1,225 dpm 16 hours after collection, calculate the half-life and activity of the beta emitter other than radon or thoron daughters.
2. Sketch a continuous air sampler that would monitor alpha activity and trigger an alarm at a certain set level.
3. Write the procedure for collecting an air sample for particulate analysis.

MATERIALS

Beta counter.
Alpha counter.
High-volume air sampler.
Low-volume air sampler.
Timer.
Glass fiber filters for both samplers.
Whatman #40 filter paper.
Membrane filter.
Analytical balance.
Chemical balance.
Platinum crucible.
Muffel furnace.
Drying oven.
Hydrochloric acid, concentrated, 1N, 2N, 6N, 8N, 9N.
Hydrofluoric acid, concentrated (48%), 1.5N.
Sodium carbonate, anhydrous powder, 0.1N, 3N.
Acetone.
Electrodeposition unit.
Stainless steel planchets.
Nickel plated copper planchets.
Copper planchets.
Aluminum nitrate.
Lanthanum nitrate.
Hydroxylamine hydrochloride, crystals.
pH meter.
Nitric acid, concentrated, 1N, 8N, red fuming.
Potassium hydroxide, 2N, 8N.
Sodium hypochlorite, analytical grade.
Sodium nitrite, 2N.
2-thenoyltrifluoroacetone, 5.6% w/v in xylene.

Ammonium oxalate, 0.032N.
Ethyl alcohol, absolute (100%).
Ion-exchange column.
Dowex 1-X4 ion-exchange resin, chloride form, 50–100 mesh.
Acetic acid, glacial, 6N.
Ammonium acetate.
Centrifuge.
100-ml centrifuge tubes, polyethylene.
Buchner funnel, polyethylene.
Magnetic stirrer with stirring bars.
Vacuum filtration apparatus.
Ammonium hydroxide, concentrated, 6N.
Barium nitrate.
Strontium nitrate.
Yttrium nitrate.
Diethyl ether.
Hydrogen peroxide, 30%.
Oxalic acid, 2N.
Sodium chromate, 0.6N.
Sodium hydroxide, 10N.
Glassine envelopes to fit filters.

PROCEDURES

Note: All radiochemical procedures except weighings must be conducted in a fume hood.

PROCEDURE 1: FILTER SAMPLING FOR AIRBORNE PARTICULATES (LOW RATE)

1. Assemble a low-volume air sampler with glass fiber filter.
2. Place sampler in an appropriate location; turn it on.
3. Record the time in Data Table 20-3-1.
4. Adjust air flow to 25 liters per minute.
5. Record flow rate.
6. Allow to run for 3 hours.
7. Record air flow rate after 3 hours.
8. Turn off sampler.
9. Remove filter from sampler; place filter in a glassine envelope.
10. Quickly carry envelope to counting room.
11. Remove filter from glassine envelope; count for alpha and beta activity.
12. Save filter in glassine envelope for continued analysis.
13. Record data in Data Table 20-3-1.

PROCEDURE 2: FILTER SAMPLING FOR AIRBORNE PARTICULATES (HIGH RATE)

1. Assemble a high-volume air sampler with glass fiber filter.
2. Place sampler in an appropriate location; turn it on.
3. Record time and air flow rate in Data Table 20-3-2.
4. Collect sample for 35 minutes.
5. Record time and final air flow rate.
6. Turn off sampler.
7. Remove filter; place filter in a glassine envelope.
8. Carry filter into preparation room.

9. Remove filter from envelope; cut a disc 5 cm in diameter from filter.
10. Replace remainder of filter into envelope; save for future analysis.
11. Place disc on a planchet; carry into counting room.
12. Count sample for alpha and beta activity.
13. Save this filter disc for future analysis.
14. Record data in Data Table 20-3-2.

PROCEDURE 3: USE OF CELLULOSE FIBER FILTERS (LOW RATE)

1. Repeat Procedure 1, using a Whatman #40 filter disc instead of a glass fiber filter. NOTE: Have instructor spike this filter with a milligram or two of uranium compound.
2. Record data in Data Table 20-3-3.

PROCEDURE 4: USE OF A MEMBRANE FILTER

1. Repeat Procedure 1, using a membrane filter having 0.2-micron diameter pores. NOTE: Have instructor spike this filter with 0.1 microcurie of ^{90}Sr.
2. Record data in Data Table 20-3-4.

PROCEDURE 5: DETERMINATION OF ACTIVITY AND CONCENTRATION OF CONTAMINATION

NOTE: The three samples collected with the low-volume sampler are to be used in this exercise. Provision must be made to count each sample at the required time. Any deviation requires prior approval of the instructor.

1. Count each sample for alpha and beta activities 6 hours after the sample is collected.
2. Record time and activity.
3. Wait 10 hours after counting each sample; count it again.
4. Record time and activity in Data Table 20-3-5.
5. From Data Tables 20-3-1, 20-3-3, and 20-3-4, calculate the first count ratio for each sample.
6. Record first count ratios; list type of suspected contamination on each filter.
7. Calculate activity on each filter and the air concentration of the contaminant on each filter. Use activity fraction and activity ratio.
8. Record in Data Table 20-3-5.

PROCEDURE 6: SEPARATION AND DETECTION OF PLUTONIUM

NOTICE: The probability of having obtained plutonium in the air sample is quite small. The instructor should spike the sample with a trace of plutonium if it is available. Use the sample collected for the glass fiber filter.

1. Weigh filter; place it in a platinum crucible.
2. Cover filter with three times its own weight of anhydrous sodium carbonate.
3. Place sample and crucible in a muffel furnace; fuse the sample at 950 ± 20°C for 30 minutes.
4. Remove crucible; allow it to cool.

5. Add 50 ml of distilled water; heat on a hot plate until liquid just begins to boil.
6. Transfer liquid and solid to a beaker.
7. Neutralize solution with concentrated HCl.
8. Estimate volume of solution; add sufficient HCl to make the solution 2N in HCl.
9. Filter the residue, using Whatman No. 40 filter paper. Collect and save filtrate. Wash residue 2 times with 5-ml portions 2N HCl and 2 times with 5-ml portions of distilled water. Add washings to filtrate.
10. Transfer filter paper to a platinum crucible; ignite in a furnace.
11. Cool crucible; add 10 ml of concentrated HF; and evaporate to dryness on a hot plate.
12. Add 5 ml of 6N HCl; evaporate to dryness on a hot plate.
13. Repeat Step 12.
14. Dissolve the chloride salts in the crucible with 2N HCl; combine with the filtrate in Step 9.
15. Transfer to a volumetric flask; dilute to 100 ml with 2N HCl.

NOTICE: The preceding is the procedure for the preparation of a sample collected on a glass fiber filter. The following procedures are for separation and electrodeposition of plutonium.

1. Pipette an aliquot of solution containing at least an estimated 1 picocurie of plutonium into a 100-ml polyethylene container. (A smaller sample may be used, but it does not function very well. A 10-picocurie or larger sample is preferred.) Dilute to 75 ml with 2N HCl.

2. Add 1 g of hydroxylamine hydrochloride and 62 mg of lanthanum nitrate hydrate, stir, and let stand for 10 minutes.
3. Add 10 ml of concentrated hydrofluoric acid; let stand for 10 minutes.
4. Transfer to polyethylene centrifuge tubes; centrifuge for 10 minutes at 2000 rpm. Decant and discard the supernate.
5. Add 10 ml of 1.5N HF, mix, and centrifuge for 10 minutes at 2000 rpm. Discard the supernate.
6. Dissolve precipitate in 30 ml of 0.45N aluminum nitrate. Add 0.25 ml of 2N $NaNO_2$ and stir.
7. Transfer solution to a 125-ml separatory funnel. Wash centrifuge tubes twice with 5-ml portions of distilled water, adding washings to solution in the separatory funnel.
8. Add 10 ml of TTA reagent (5.6% w/v 2-thenyltrifluoroacetone in xylene); shake for 20 minutes. Allow phases to separate for 5 minutes, then draw off and discard the aqueous phase.
9. Add 20 ml of distilled water to funnel; shake for 10 minutes. Allow phases to separate, and discard the aqueous phase.
10. Repeat Step 9.
11. Repeat Step 9, using 10 ml of distilled water.
12. Add 10 ml of 8N HCl; shake for 20 minutes to extract plutonium. Allow phases to separate for 5 minutes, and collect the acid phase in a 50-ml beaker. Repeat with 5 ml of 8N HCl, and combine the acid phases.
13. Evaporate the acid solution to a volume of 1 to 2 ml. NOTICE: DO NOT EVAPORATE TO DRYNESS.

14. Neutralize remaining solution with 8N KOH. DO NOT EXCEED A pH OF 9.5. Pu^{+4} will precipitate from a highly alkaline solution.
15. Add 2 ml of sodium hypochlorite solution and 5 ml of 2N KOH. Evaporate to a volume of 5 ml.
16. Cool and transfer to a Teflon electrodeposition cell equipped with a stainless steel planchet.
17. Electroplate for 5 hours at 7.5 volts and 200 milliamps. Be sure to remove anode before turning off power.
18. Pour off plating solution; rinse the cell thoroughly with distilled water.
19. Remove planchet, rinse with distilled water, drain, then flame to cherry red over a burner.
20. Cool planchet; count on an alpha counter.
21. Calculate plutonium activity per cubic meter of air sampled. Remember that only half the sample in the volumetric flask was used.
22. Record activity level in Data Table 20-3-6.

PROCEDURE 7: SEPARATION AND DETECTION OF URANIUM

Use the cellulose fiber filter, previously spiked with uranium, for this analysis.

1. Place cellulose filter containing the sample in a platinum crucible; ignite in a muffel furnace.
2. Cool crucible; add 10 ml of hydrofluoric acid.
3. Evaporate to dryness on a hot plate.
4. Add 5 ml of 6N HCl; evaporate to dryness on hot plate.
5. Repeat Step 4.

6. Dissolve remaining chlorides in 2N HCl, quantitatively transfer to a volumetric flask (100 ml), and dilute with 2N HCl.

NOTICE: The preceding is the procedure for the preparation of a sample collected on a cellulose fiber filter. The following procedures are for separation and electrodeposition of uranium.

1. Pipette 50 ml of the solution to be analyzed into a 250-ml beaker. Add sufficient concentrated HCl to make the solution 6N in HCl, then dilute to 200 ml with 6N HCl.
2. Transfer acid solution to a separatory funnel attached to a previously conditioned ion-exchange column containing Dowex 1-X4 500–100 mesh anion-exchange resin. Condition the resin by flowing 500 ml of 6N HCl through at 2 ml/min. Let solution flow through system at 2 ml/min. Discard effluent.
3. Place 200 ml of 9N HCl in separatory funnel; wash resin at a flow rate of 2 ml/min.
4. Place 150 ml of 1N HCl in separatory funnel; flow through at 2 ml/min, eluting the uranium. Collect eluate in a 250-ml beaker.
5. Evaporate eluant to dryness on a hot plate.
6. Add 5 ml of 1N HNO_3; evaporate to dryness.
7. Repeat Step 6.
8. Add 10 ml of 0.032N ammonium oxalate; police the beaker.
9. Repeat Step 8.
10. Repeat Step 8, using 5 ml of ammonium oxalate solution.
11. Quantitatively transfer solution to a Teflon electrodeposition cell equipped with a nickel plated copper planchet.

12. Electroplate for at least 17 hours at 7 volts and 40 milliamps with the platinum anode stirring at approximately 200 rpm.
13. Remove planchet; rinse with distilled water, then with absolute ethyl alcohol. Flame over a burner, cool, and alpha count.
14. Calculate uranium activity per cubic meter of air sampled.
15. Record data in Data Table 20-3-6.

PROCEDURE 8: SEPARATION AND DETECTION OF RADIOSTRONTIUM

Use the membrane filter, previously spiked with radiostrontium.

1. Place membrane filter in a platinum crucible; add 5 ml of acetone to dissolve the membrane.
2. After membrane is dissolved, evaporate solution to dryness.
3. Ignite residue in a muffel furnace.
4. Cool crucible; add 10 ml of concentrated HF.
5. Evaporate to dryness on a hot plate.
6. Add 5 ml of 6N HCl; evaporate to dryness on a hot plate.
7. Repeat Step 6.
8. Dissolve remaining chlorides in 2N HCl. Quantitatively transfer to a 100-ml volumetric flask, and dilute with 2N HCl.

NOTE: The preceding is the procedure for the preparation of a sample collected on a membrane filter. The procedure for separation and oxalate precipitation of radiostrontium follows.

1. Pipette 50 ml of solution to be analyzed into a 250-ml beaker. Add 1 ml of a solution containing 64.75 mg of strontium nitrate tetrahydrate and 1 ml of a solution containing 38.05 mg of barium nitrate per ml of solution. Stir for 15 minutes on a magnetic stirrer.
2. Dilute to 200 ml with distilled water; adjust to pH 8 with 10N NaOH.
3. Add 1 g of sodium carbonate; stir for 15 minutes. Allow precipitate to settle for 3 hours.
4. Decant and discard supernatant solution. Transfer precipitate to a centrifuge tube, and centrifuge. Discard supernatant.
5. Wash beaker 3 times with 10-ml portions of water, transferring the wash to the centrifuge tube. Stir, centrifuge, and discard supernate.
6. Dissolve precipitate in 10 ml of 8N HNO_3; transfer to a 250-ml beaker. Evaporate slowly to near dryness on a hot plate.
7. Add 23 ml of distilled water and 77 ml of fuming HNO_3. Stir for 15 minutes on a magnetic stirrer.
8. Allow to stand for 5 minutes. Then filter with suction through a glass fiber filter; suction dry and discard the filtrate.
9. Rinse beaker 3 times with 10-ml portions of distilled water, pouring the washings through the filter, dissolving the precipitate and collecting the solution in a clean 250-ml beaker.
10. Evaporate solution slowly to dryness.
11. Repeat Steps 7 through 10.
12. Transfer solution to a centrifuge tube; adjust the volume to 20 ml.

13. Adjust pH to 8 with concentrated NH_4OH to check for presence of phosphate. If no precipitate forms, adjust pH to 2 with 1N HNO_3. If a precipitate forms, repeat Steps 7 through 10.

14. Add 1 ml of an yttrium carrier solution containing 43.08 mg of yttrium nitrate hexahydrate per ml; heat in a 90°C water bath for 5 minutes.

15. Adjust pH to 8 with concentrated NH_4OH; heat again in the water bath for 5 minutes. Cool to room temperature; allow to stand for 5 minutes.

16. Centrifuge for 10 minutes; decant into a clean centrifuge tube.

17. Dissolve precipitate in a minimum volume of concentrated HCl. Dilute to 15 ml with distilled water, and repeat Steps 15 and 16.

18. Combine decantates into a single centrifuge tube; discard any precipitate. Adjust volume to 25 ml with distilled water; add 1 ml of 6N acetic acid and 2 ml of 6N ammonium acetate. Adjust pH to 5.5 with 6N NH_4OH.

19. Heat solution in water bath for 2 minutes; add 1 ml of 0.6N sodium chromate dropwise, with constant stirring. Digest in water bath for 30 minutes, cool to room temperature, and centrifuge for 10 minutes.

20. Decant the supernate into a polyethylene container; adjust pH to 2 with 1N HNO_3. Add 1 ml of yttrium carrier, stopper, and store for 2 weeks.

21. After 2 weeks, transfer solution to a centrifuge tube; adjust volume to 25 ml with distilled water.

22. Adjust pH to 8 with concentrated NH_4OH. Stir, then add 3 drops of 30% hydrogen peroxide. Heat for 10 minutes in the hot water bath, to precipitate yttrium hydroxide, $Y(OH)_3$.

23. Cool to room temperature; centrifuge for 10 minutes. Decant into a 100-ml beaker. NOTICE: Record in Data Table 20-3-6 the time of decantation as the beginning of yttrium-90 decay in the precipitate and yttrium-90 regrowth in the total strontium supernate.
24. Dissolve precipitate in a few drops of concentrated HCl; dilute to 25 ml with distilled water.
25. Repeat Steps 22 and 23, combining supernatent liquids for total radiostrontium determination.
26. Add 6N HCl to precipitate, one drop at a time, until all the precipitate just dissolves. Dilute to 25 ml with distilled water; add 5 ml of 2N oxalic acid; adjust the pH to 1.5 with 6N NH_4OH.
27. Heat in a water bath for 30 minutes. Cool to room temperature; filter with suction through a previously weighed glass fiber filter.
28. Wash centrifuge tube and precipitate with 10 ml of distilled water. Dry the precipitate by rinsing 3 times with 10-ml portions of ABSOLUTE ethyl alcohol and 3 times with 10-ml portions of diethyl ether. Continue suction for 2 minutes.
29. Weigh after a delay of 10 minutes; calculate recovery of the yttrium carrier by dividing the weight of the yttrium oxalate precipitate by the weight of yttrium oxalate obtained from 1 ml of carrier solution.
30. Beta count yttrium-90 in precipitate; record the time of counting.
31. Heat the supernate from Step 25 in a water bath for 15 minutes. Slowly add 10 ml of 3N sodium carbonate with continuous stirring. Heat in a water bath for 30 minutes.

32. Cool to room temperature; filter with suction through a pre-weighed glass fiber filter, washing container and precipitate with 10 ml of 0.1 N sodium carbonate.

33. Dry in a drying oven at 110°C for 20 minutes; cool in a desiccator for 10 minutes. Weigh and calculate recovery of the strontium carrier as strontium carbonate.

34. Beta count sample activity. Record time of counting.

35. Calculate according to the following equations:

$$^{90}\text{Sr activity (pCi/m}^3\text{)} = \frac{A}{B \times C \times D \times E \times F \times G \times H}$$

where:

A = net count rate of yttrium-90.

B = total air sample volume (m^3).

C = fraction of total sample analyzed.

D = counter efficiency for counting yttrium oxalate/yttrium-90 mounted on a glass fiber filter (cpm/pCi).

E = chemical recovery of yttrium (from Step 29).

F = correction factor $e^{-\lambda t}$ for yttrium-90 decay, where t is the time from separation of yttrium (Step 23) until the time of counting (Step 30).

G = strontium carrier recovery.

H = correction factor $1-e^{-\lambda t}$ for yttrium growth, where t is the time from the first separation of yttrium from strontium to the separation following storage period.

$$^{89}\text{Sr activity (pCi/m}^3\text{)} = \frac{1}{B \times C}\left[\frac{A}{D \times E \times F} - G(H + I \times J)\right]$$

where:

A = net beta count rate of the total radiostrontium (cpm).

B = correction factor $e^{-\lambda t}$ for strontium-89 decay, where t is the time from sample collection until the time of counting.

C = counting efficiency for counting strontium-89 as strontium carbonate mounted on a glass fiber filter (cpm/pCi).

D = strontium carrier recovery (from Step 33).

E = total air volume (m^3).

F = fraction of total sample analyzed.

G = strontium-90 activity as computed from yttrium-90 activity (pCi/m^3).

H = counter efficiency for counting strontium-90 as strontium carbonate on a glass fiber filter (cpm/pCi).

I = counter efficiency for counting yttrium-90 as yttrium oxalate mounted on a glass fiber filter (cpm/pCi).

J = correction factor $1-e^{-nt}$ for yttrium-90 growth, where t is the time from separation of the strontium (Step 23) until the time of counting (Step 34).

36. Record activities in Data Table 20-3-6.

DATA TABLE 20-3-1
LOW-RATE SAMPLING

	Time	Flow 1/min
Start	__________	__________
Stop	__________	__________

Total time of sample __________ hours

Average flow rate __________ liters/minute

Total volume of sample __________ liters

Radiation Type	Net Activity dpm	Elapsed Time After Sampling
alpha	____________	_____________
beta	____________	_____________

DATA TABLE 20-3-2
HIGH-RATE SAMPLING

	Time	Flow 1/min.
Start	__________	__________
Stop	__________	__________

Total time of sample __________

Average flow rate __________ liters/minute

Total volume of sample __________ liters

Radiation Type	Net Activity dpm	Elapsed Time After Sampling
alpha	____________	______________
beta	____________	______________

DATA TABLE 20-3-3

LOW-RATE SAMPLING – CELLULOSE FIBER FILTER

	Time	Flow l/min
Start	__________	__________
Stop	__________	__________

Total time of sample __________ hours

Average flow rate __________ liters/minute

Total volume of sample __________ liters

Radiation Type	Net Activity dpm	Elapsed Time After Sampling
alpha	____________	______________
beta	____________	______________

DATA TABLE 20-3-4
LOW-RATE SAMPLING – MEMBRANE FILTER

	Time	Flow l/min
Start	__________	__________
Stop	__________	__________

Total time of sample __________

Average flow rate __________ liters/minute

Total volume of sample __________ liters

Radiation Type	Net Activity dpm	Elapsed Time After Sampling
alpha	__________	__________
beta	__________	__________

DATA TABLE 20-3-5

ACTIVITY AND CONCENTRATION OF CONTAMINATION

Sample	Activity of filter	dpm		
		at time of sample	after 6 hours	after 16 hours
G-F	A			
	A			
Cellu	A			
	A			
Memb	A			
	A			

Sample	First Count Factor Γ	Suspect Contamination	Total Activity dpm	Total Air Volume m^3	Contamination Concentration dpm/m^3
G-F					
Cellu					
Memb					

DATA TABLE 20-3-6

RADIOCHEMICAL SEPARATIONS

Sample	Original Activity on Filter dpm	Activity on Planchet dpm	Calculated Total Activity uCi	Contamination in Air uCi/m^3
Plutonium				
Uranium				
Radiostrontium	pCi/m^3			
^{90}Sr	__________			
^{89}Sr	__________			

REFERENCE MATERIALS

APHA-AWWA-WPCF. Standard Methods for the Examination of Water and Wastewater. 13th ed. New York: American Public Health Association, Inc., 1971.

Health and Safety Laboratory. EML Procedures Manual, HASL-300, Revisions to August 1979. New York: U. S. Energy Research and Development Administration, 1979.

TEST

1. A sample of particulates from an air sample was found to have the following activities at the times given:

Radiation Type	Activity in dpm		
	at time of collection	after 6 hours	after 16 hours
alpha	47,000	20,455	7,407
beta	77,000	48,533	21,837

What is the half-life of the alpha emitter and of the beta emitter?

2. Sketch a schematic diagram of a continuous air particulate sampler that continuously will record activity levels and will sound an alarm if the level exceeds a set point.

3. Given an air sample of 970 m^3 and a total activity of 0.14 μCi, what is the contamination level in pCi/m^3?

4. Write the procedure for the collection of a low-volume sample on a glass fiber filter.

5. Write the procedure for the collection of a sample on a glass fiber filter, using a high-volume sampler.

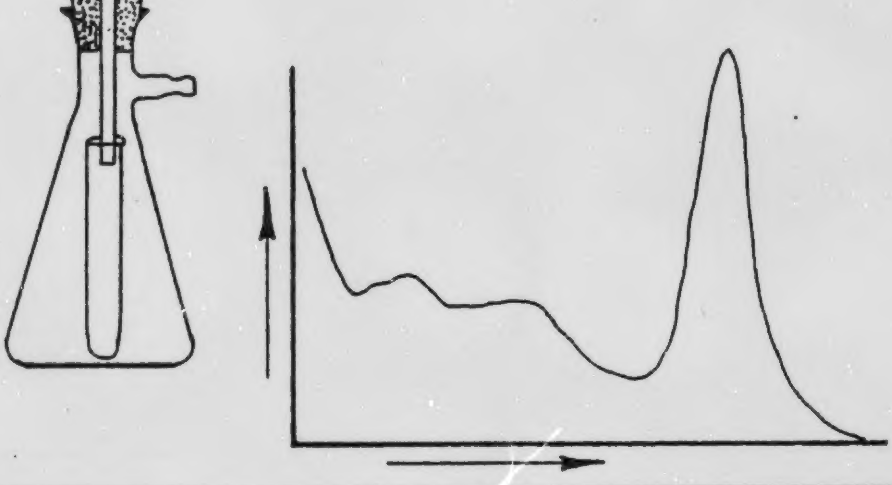
NUCLEAR TECHNOLOGY

MODULE 20-4

AIR MONITORING TECHNIQUES - GASEOUS

RADIATION MONITORING
TECHNIQUES (RADIOCHEMICAL)

COURSE 20



CORD CENTER FOR OCCUPATIONAL RESEARCH AND DEVELOPMENT

COURSE MODULES

COURSE 20 RADIATION MONITORING TECHNIQUES (RADIOCHEMICAL)

MODULE 20-1	Radiation Sample Preparation — Liquid
MODULE 20-2	Radiation Sample Preparation — Solid
MODULE 20-3	Air Monitoring Techniques — Particulate
⇨ MODULE 20-4	Air Monitoring Techniques — Gaseous
MODULE 20-5	Water Monitoring Techniques — Dissolved Solids
MODULE 20-6	Water Monitoring Techniques — Suspended Solids
MODULE 20-7	Radiation Monitoring of Biological Samples

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INTRODUCTION

Some radionuclides are in the atmosphere in the form of gases. These potentially harmful gases must be sampled in order that their concentrations can be determined. Such gases exist in, and may be released from, certain nuclear facilities. In these facilities, gas samples may be drawn from cylinders, piping, ducts, etc.

Sampling and analysis techniques for gaseous radionuclides are not as advanced or sophisticated as those for particulate matter. There is no simple device for the separation of gases. The procedures depend upon physical and chemical properties and involve such techniques as adsorption, absorption, and diffusion. This module discusses techniques and procedures for the monitoring of gaseous radionuclides.

MODULE PREREQUISITES

The student should have completed Course 1, "Radiation Physics"; Course 11, "Radiation Detection and Measurement"; and the previous modules of this course.

OBJECTIVES

Upon completion of this module, the student should be able to:

1. Collect a gas sample by adsorption.
2. Collect a gas sample by absorption.
3. Collect a grab sample of a gas.
4. Determine the half-life of a normal background gaseous sample using a GM counter.

5. Calculate activity per unit volume of a gaseous sample.
6. Describe the types of detectors used for the counting of gaseous samples.

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SUBJECT MATTER

Several radionuclides exist in the atmosphere as gases. Both gaseous fission products and neutron activation products contribute to gaseous contamination of the atmosphere. Specifically, tritium, iodine-131, carbon-14, and isotopes of the noble gases — for example, argon, krypton, and xenon — are of concern to the nuclear radiation industry.

Generally, sampling methods and measurement techniques are less satisfactory than those for particulates. There is not a simple device, such as a filter, that will separate a radioactive gas from air. Separation procedures depend upon the physical and chemical properties of the isotopes and involve such techniques as diffusion, adsorption, and absorption.

GASEOUS SAMPLE COLLECTION TECHNIQUES

The three most common techniques for the collection of gaseous material samples are adsorption, absorption, and grab sampling. Each of these has its merits and should be considered for specific applications.

ADSORPTION

Adsorption techniques for radioactivity measurements currently are employed primarily for the monitoring of gaseous releases from reactors and spent-fuel reprocessing operations. The procedure, in conjunction with filtration, can determine environmental levels of radioiodine and radon.

Basic Principles

Adsorption is the phenomenon of attraction, concentration, and retention of gases, liquids, and solutes at a boundary surface. The boundary surface may be the interface between a gas and a liquid, a liquid and a liquid, a gas and a solid, a liquid and a solid, or a solid and a solid. The gas-and-solid interface has proved useful in the sampling of gaseous radioactive products.

The solid adsorbent is composed of a crystal lattice structure whose attractive forces of the surface atoms are unsatisfied. This unsatisfied condition results in the attraction and retention of gases or other substances that come in contact with the surface of the lattice. This surface concentration of the material is the adsorption process. The substance that provides the surface and does the attracting is known as the "adsorbent," and the substance attracted is known as the "adsorbate."

When applied to gas collection techniques, adsorption commonly is utilized for the collection of a specific gas or combination of gases. Typically, the process involves the passing of a gas stream through a container containing an adsorbent such as activated charcoal, alumina, or silica gel. The atomic and molecular forces bind the gas to the adsorbent; and the gas remains physically and chemically unchanged, provided that condensation does not occur. A filter adsorption train is illustrated in Figure 20-4-1. The gas may or may not be removed from the adsorbent for analysis, depending upon the counting system. Removal of the gas from the adsorbent may be accomplished by the passing of an inert carrier gas through the system, application of heat, chemical treatment, or a combination of these.

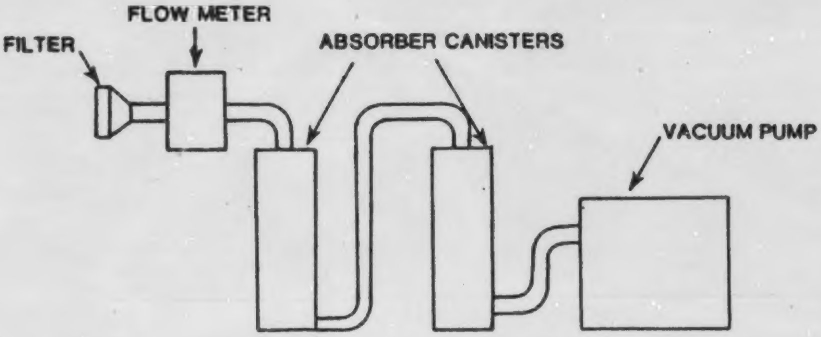


Figure 20-4-1. A filter adsorber train.

Adsorption must not be confused with absorption. Adsorption retains the material on the surface of the adsorbent. In absorption, the material not only is held on the surface, but passes through the surface and is distributed throughout the absorption medium.

Types of Adsorption

Research into the processes of adsorption of gases on various solid surfaces has revealed that the operating forces are not the same in all cases. Two types of adsorption processes currently are recognized: (1) activated, or chemical, adsorption; and (2) physical, or van der Waals, adsorption.

Chemical, or activated, adsorption provides a much stronger binding of the gas molecule to the surface than does physical adsorption. The process involves the combination of the gas molecule with the adsorbent to form a surface compound. This type of adsorption resembles chemical bonding; hence, the name chemical adsorption.

The attractive forces in physical adsorption consist of van der Waals forces, dipole-dipole interaction, and/or electrostatic attraction. Since these forces are relatively small, the adsorption equilibrium is reversible and rapidly established. This physical adsorption is a generally occurring process, and any gas can be adsorbed to some extent. The quantity of different gases adsorbed under the same conditions is directly proportional to their critical temperature: The higher the critical temperature, the greater the quantity of gas that can be adsorbed.

Generally, considering adsorbent-adsorbate pairs, chemical adsorption is much more specific or selective than physical adsorption. This process is slower because of the requirement for displacement or selection of the molecules at the reaction site. Chemical adsorption is enhanced by elevated temperatures. A major disadvantage is that chemical adsorption usually is limited to the formation of a single molecular layer on the adsorbent surface.

Physical adsorption proceeds well at low temperatures, and the adsorbed layer may be several molecules thick. Physical adsorption is the major process used in the collection of radioactive gases.

Variables that Affect Adsorption

The amount of a particular gas that can be adsorbed onto a given quantity of adsorbent depends upon several factors, which include —

- The concentration of the gas.
- Total surface area of the adsorbent.
- Temperature of the system.
- Presence of other gases.
- Characteristics of the gas.
- Size and shape of the pores in the adsorbent.
- Surface polarity and chemical reactivity of the adsorbent.
- Reaction time (time during which the gas is in contact with the adsorbent).

Characteristics of Adsorbents

Common adsorbents are granular in form and are supported in a column through which the gas to be sampled is drawn. Typically, the adsorbents have the capacity to adsorb from 8 to 40 percent of their weight. The ideal adsorbent should be of such size and form to offer practically no resistance to gas flow. It should have a high adsorptive capacity; should be specific, inert; resistant to breakage, deterioration, and corrosion; easily activated; and should provide easy release of the adsorbate. Regrettably, no adsorbent possesses all these characteristics.

Typical Adsorbents

Typical adsorbents for physical adsorption may be classified according to their degree of polarity. Activated carbon, being nonpolar, usually is listed first, and may be used as a benchmark for comparison to other adsorbents.

The majority of the remaining commercially important adsorbents, other than carbon derivatives, are simple or complex oxides. As a general rule, they are strongly polar and show a high degree of selectivity in the separation of some gases. Three common adsorbents are activated carbon, silica gel, and activated alumina.

Adsorption techniques primarily are aimed at the monitoring of gaseous releases from reactors and fuel reprocessing operations. The procedures may be both qualitative and quantitative. Iodine-131 routinely is monitored by adsorption onto activated carbon. Additionally, the noble gases – argon, xenon and krypton – can be adsorbed on activated charcoal. Since each noble gas has a specific affinity for activated carbon, each can be separated by chromatographic adsorption (preferential adsorption of gases in an ascending molecular-weight sequence onto a solid adsorbent).

ABSORPTION

Absorption by a liquid solution is probably the most widely used gas sampling technique. The popularity of this technique is due to the great variety of analytical methods available for investigation of the resulting solutions, procedure reproducability, and the relative ease with which reliable data can be obtained.

Types of Absorption

The technician can accomplish gas-liquid absorption sampling either by dissolving the gas in a liquid or by reacting the gas with a liquid. The collecting liquid may change physically and/or chemically during the collection process.

Physical or dissolution absorption involves the physical solution of the gas in a liquid. The process usually is reversible and may be simply a step performed in order to concentrate the gas. Often, however, the solution is used in the final analysis. The ideal solvent should be relatively nonvolatile, and should be inexpensive, noncorrosive, stable, nonviscous, nonconforming, and nonflammable.

Chemical absorption, in contrast to physical absorption, involves the reaction of a liquid absorbent with the gas to produce a nonvolatile product. These reactions usually are irreversible without further chemical treatment. The choice of a chemical absorbent is influenced by solubility of the gas, by reactive properties of gas and absorbent, and by analytical methods to be used.

Absorption Devices

A large variety of devices have been utilized as absorption samples for atmospheric gases. One of the simplest and most common is an ordinary gas washing bottle, which contains the absorbent, equipped with a dispersion tube for introduction of the gas into the solution. Figure 20-4-2 is an illustration of the gas in the solution. Gas flows through the opening in the tube into the absorbent solution in the container.

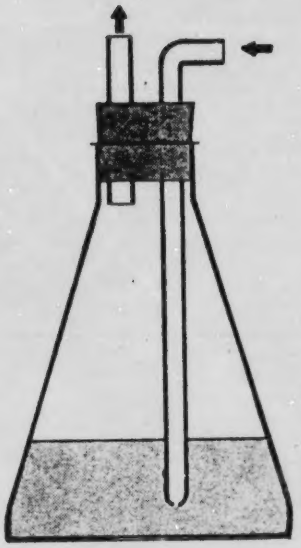
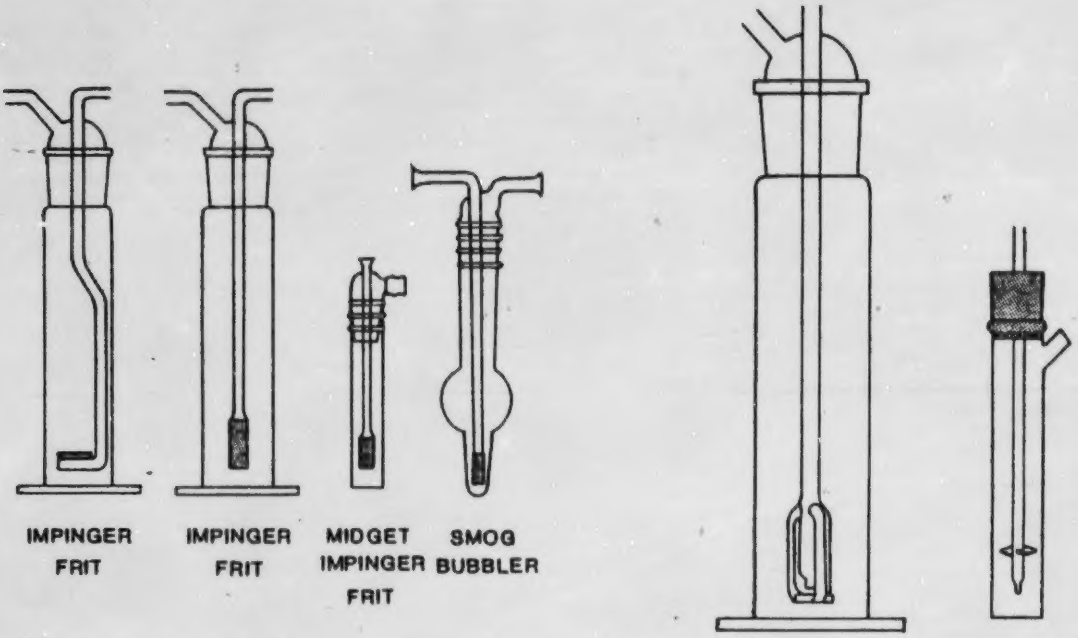


Figure 20-4-2. A simple gas absorption bottle.

There are two other common types of gas absorption devices: (1) fritted-glass absorbers and (2) impingers. The fritted-glass absorbers differ from the simple device only in that the gas dispersion tube is fitted with a glass frit that has very fine pores. The frit decreases the size of the bubbles and increases the efficiency of absorption.

Impingers are designed in such a manner that the gas flow impinges on a solid surface to help break up the bubbles. Investigation has shown that the fritted-glass absorber is, by far, the most efficient of the three absorbers. Figure 20-4-3 illustrates fritted-glass absorbers and impingers. One disadvantage of fritted-glass absorbers and impingers is that they are so fragile that they are easily broken.



FOUR TYPES OF FRITTED-GLASS ABSORBERS

TWO TYPES OF IMPINGERS

Figure 20-4-3. Fritted-glass absorbers and impingers.

Radioactive Gas Absorption

Absorption sampling techniques have been found to be very effective for the following radioactive gases: ^{14}C in the form of carbon dioxide and ^{131}I as molecular iodine.

Because atmospheric ^{14}C from both natural and man-made sources eventually becomes carbon dioxide, monitoring of ^{14}C requires quantitative sampling of carbon dioxide. Quantitative sampling is accomplished either by absorption into an alkaline solution to produce a soluble carbonate, or by direct precipitation as a carbonate from a basic alkaline earth solution. Either method produces a collection efficiency of 90 percent or better under optimum conditions.

The procedure for direct precipitation is quite simple, and is depicted in Figure 20-4-4. Four absorption bottles, each containing 2 liters of a saturated barium hydroxide solution, are connected in series to a suction pump. As the gases are drawn through the barium hydroxide, barium carbonate precipitates. Most of the barium carbonate precipitates in the first absorber, and only small quantities collect in the fourth absorber. After the sample is collected, the barium carbonate can be concentrated into a smaller volume and counted directly or treated chemically before counting.

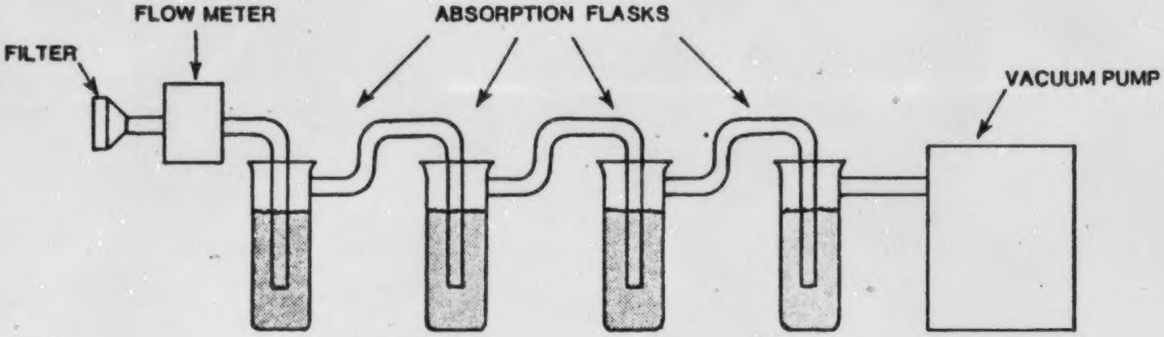


Figure 20-4-4. A filter absorber train.

Absorption as a soluble carbonate is accomplished by bubbling the carbon dioxide through a solution of potassium hydroxide or sodium hydroxide. The carbon dioxide may be regenerated from the solution by addition of acid. The liberated carbon dioxide then may be subjected to various chemical treatments before analysis.

Absorption techniques are widely used for monitoring gaseous ^{131}I. Actually, since there is considerable uncertainty about quantities of gaseous and particulate iodine, absorption techniques often are used in conjuction with filtration techniques.

Gaseous ^{131}I usually is absorbed in an alkaline solution, chemically changed, separated, purified, and counted as radioiodine. One procedure that has considerable success involves the absorption of prefiltered gaseous radioiodine in an alkaline, thiosulfate solution to produce iodide ions. The iodide then is oxidized to free iodine and extracted in to chloroform, stripped into an alkaline sulfite, and gamma counted. This procedure yields a collection efficiency greater than 90 percent under optimum conditions.

Another system involves absorption of the radioiodine in a sodium hydroxide solution, addition of an iodine carrier, and precipitation with silver nitrate to produce silver iodide. The purified silver iodide then is beta or gamma counted for the ^{131}I activity.

GRAB SAMPLING

A grab sample may be either a small representative portion taken from a gross sample or a sample collected at a specified time and place within a short time interval. Gaseous grab samples usually are collected in one of the following five manners:

- Use of an evacuated container.
- Purging or displacement of the air in a container.
- Displacement of a liquid.
- Inflation of a plastic bag.
- Use of a syringe.

Each of these methods has certain advantages and disadvantages.

Evacuated Containers

Several types of evacuated containers are employed in the sampling of radioactive gases. Figure 20-4-5 illustrates two of these container types.

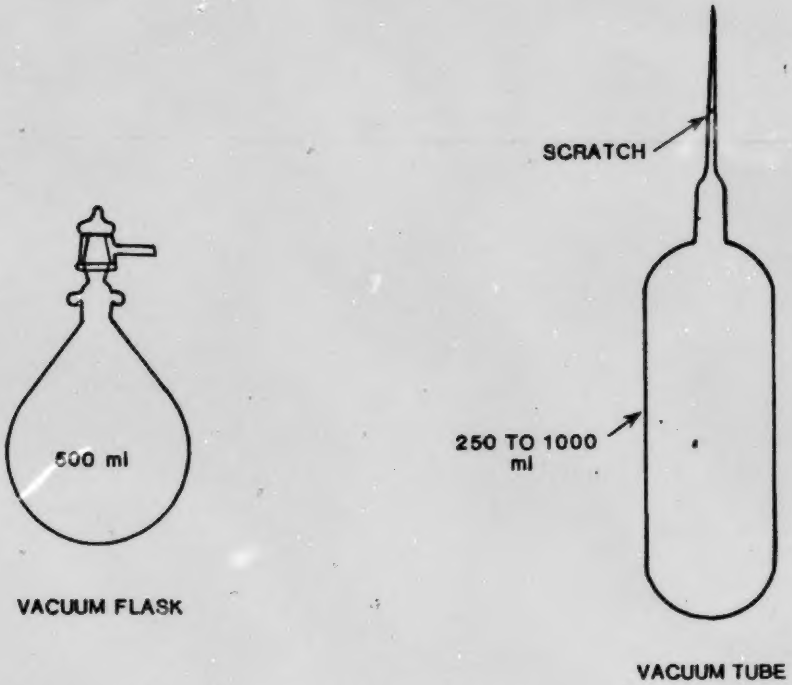


Figure 20-4-5. Evacuated containers for grab samples.

The containers are evacuated until almost all air has been removed and then sealed. The seal is opened, and the gaseous sample is allowed to fill the container until internal pressure is equal to external (atmospheric) pressure. The container opening then is resealed, and the sample is transported to the laboratory for analysis. The gas may be removed from the container for chemical treatment, or it may be counted directly in the collection vessel.

There are several advantages in the use of this type of collection device. These containers are simple to use because no pump or manometer must be taken to the sampling site. They are transported easily and require little or no special training for use in sample collection.

Disadvantages include –

- The container must be re-evacuated and sealed each time it is used.
- There is a danger of breakage or implosion.
- Containers fitted with stopcocks may have slow leaks.
- Corrections must be made for residual air volumes in the evacuated flasks.

Additionally, the presence of contamination in the residual gas in the evacuated container may require further corrections.

Purging

Air displacement or purging samplers are containers fitted with stopcocks at opposite sides. The stopcocks are opened, and the gas to be sampled is drawn through the container until the original air in the container is thoroughly purged. Volume necessary to purge the container will vary, but a volume equal to ten times the volume of the container normally is considered to be the minimum. After sampling, the container must be held in place until the stopcocks are closed and until the aspirating pump is removed. Figure 20-4-6 illustrates an example of this type of sampler.

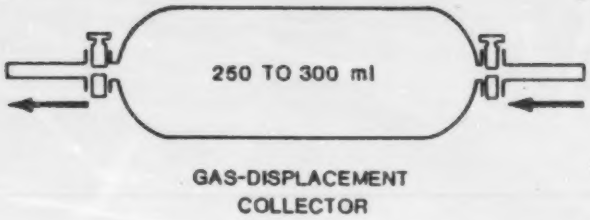
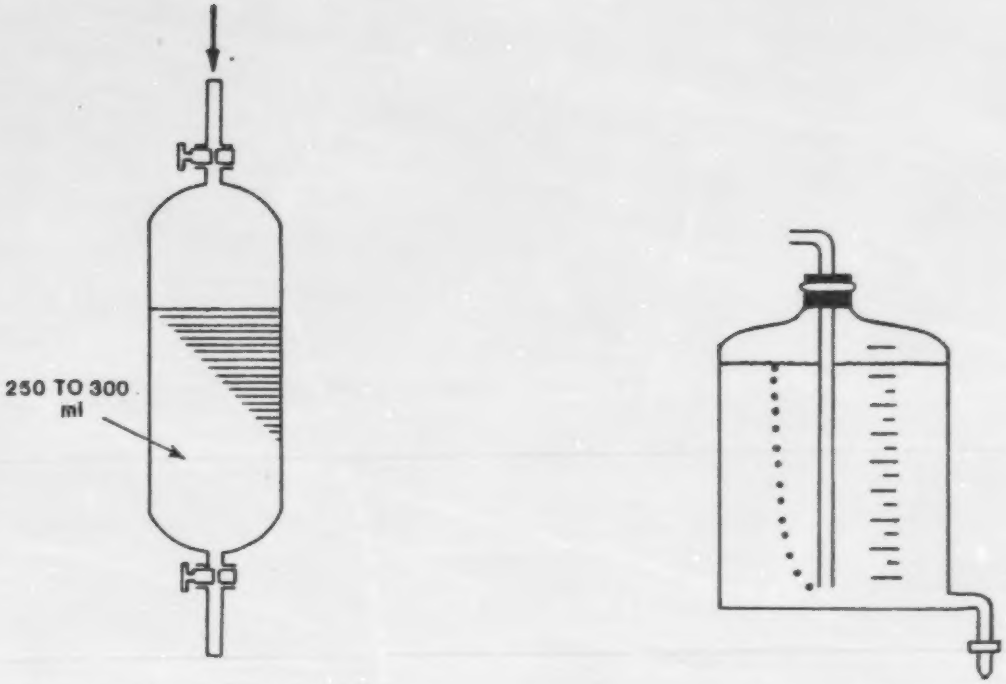


Figure 20-4-6. Gas-displacement sampling container.

Theoretically, all the old air cannot be eliminated by pumping. However, for all practical purposes, it can be. The pumping process may require a relatively long time; therefore, these samples are not "instantaneous" ones. If concentration of the radioactive material changes radically during the sampling process, the results will not provide a true indication of the actual conditions.

Displacement of Liquids

A more direct technique, which involves similar apparatus, is sample collection by liquid displacement (Figure 20-4-7). With this procedure, a liquid is allowed to drain from a container while the gas being sampled enters through another opening and fills the space left by the liquid. Any liquid that will not react with or dissolve the sample is suitable. Choice of liquid depends upon the material being sampled, but commmonly, is water, brine, water saturated with the gas to be sampled, or mercury.



LIQUID DISPLACEMENT COLLECTOR

ASPIRATOR BOTTLE

Figure 20-4-7. Liquid displacement collectors.

The containers may be the same as those used in gas displacement, or they may be of the aspirator bottle type. In either case, the liquid is allowed to drain from the lower stopcock, while the sample is drawn in through the upper stopcock. The sampling rate may be controlled by regulation of the stopcock passing the liquid, but the sampling stopcock must be full open. The container may or may not be calibrated for volume, but in either case, the method requires a minimum of equipment and no special training.

After the sample is collected, it may be counted directly or removed from the container for counting or for chemical treatment before counting.

Inflation

Still another method of gas sampling is by inflation of a plastic bag with the sample. The process is quite direct, as indicated in Figure 20-4-8. A deflated plastic bag is placed into a closed box, with only the inlet tube extending outside the box. A vacuum source is connected to an opening in the box, and the air is pumped out, allowing the bag to fill. To indicate volume of the sample, the air may be metered as it is pumped from the box. Aluminized and nonaluminized bags made of "Mylar," "Saran," and polyethylene have been found to be satisfactory, the choice depending upon the gas to be sampled.

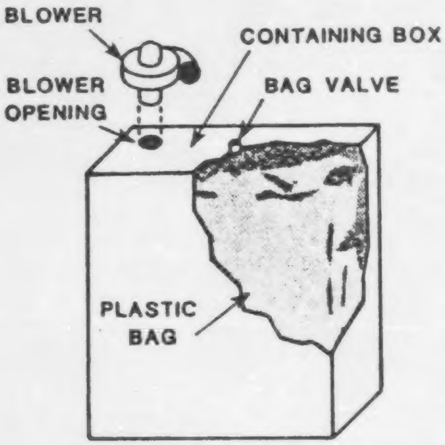


Figure 20-4-8. Grab bag sampler.

Syringes

Syringes may be used in the collection of small gaseous samples. Maximum volume is usually 100 ml, although special apparatus is available for the collection of samples up to 1 liter.

GRAB SAMPLING SUMMARY

Under certain conditions, grab sampling techniques are more useful than continuous sampling. Usually, this is true when the material to be sampled cannot be collected efficiently by absorption or adsorption, although it may be due to a lack of electricity at a collection site.

Grab sampling also is useful when concentrations vary considerably over a period of time, and when samples must be obtained at specified times. Such techniques also can provide samples for specific conditions. Additionally, grab samples require a minimum of apparatus and a minimum of training for the collection of samples.

The most serious limitation of grab sampling is that the samples collected frequently are not large enough to detect very small quantities of radioactive contaminants, except by the most sensitive techniques.

FIXED MONITORS

Some of the previously mentioned techniques may be utilized in fixed monitors, but fixed monitors usually have direct readouts or alarms, or both.

Intermittent Types

Intermittent monitors may be set up to record levels at specified times or to collect samples at definite time intervals. Radiation-level samplers normally will be set up for a specific isotope.

Intermittent sample collectors may employ any of the procedures or a combination of procedures already mentioned.

Continuous Monitors

Continuous gaseous radioactivity monitors usually are established to monitor concentrations of a specific radionuclide. They may be flow-through proportional counters such as those used to monitor tritium levels, or they may be scintillation devices connected to pulse height analyzers adjusted to monitor gamma rays from a specific radionuclide. Figure 20-4-9 is an example of an argon-41 monitoring system.

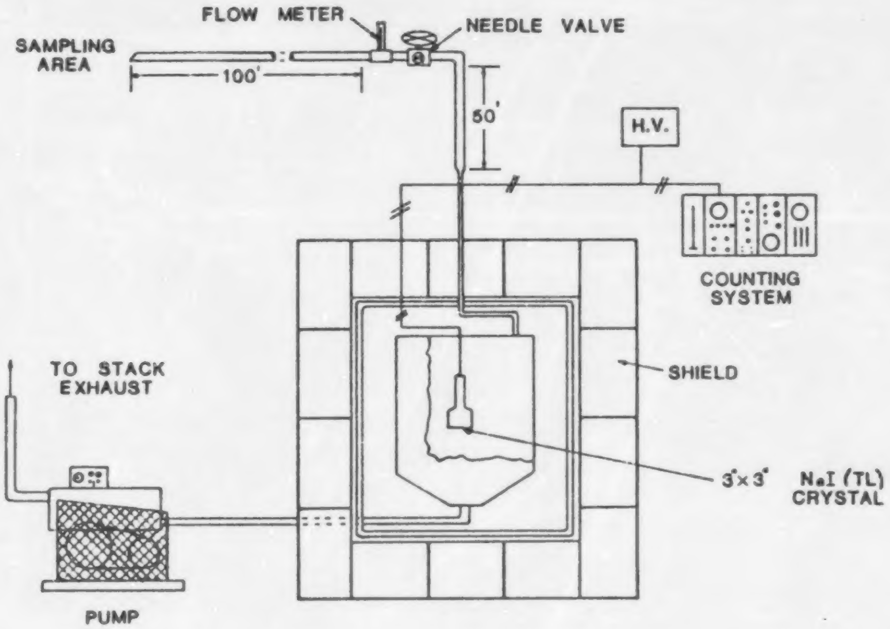

FLOW METER
NEEDLE VALVE
SAMPLING AREA
100'
50'
H.V.
COUNTING SYSTEM
SHIELD
TO STACK EXHAUST
3"×3" NaI (TL) CRYSTAL
PUMP

Figure 20-4-9. An argon-41 monitoring system.

MONITORING OF SPECIFIC GASES

Gaseous radionuclides are either decay products, activation products, or fission products. These radionuclides result from natural decay, cosmic-ray interactions, reactor operation, nuclear detonations, and the use of radionuclides.

TRITIUM

Tritium can exist in the atmosphere as hydrogen gas or as water vapor. Separation procedures are based upon the properties of hydrogen and water vapor, and involve freezing as well as chemical reactions. Tritium can be counted by a flow-through type GM counter or by a proportional counter, but usually is counted by an ionization chamber. In operation of an ionization-chamber system, compensation must be made for coincident gamma radiation.

Tritium, as a pure beta emitter, has a 0.018-MeV beta emission and a half-life of 12.5 years. It is produced in the air by cosmic particle interaction and by neutron activation. Tritium also is produced around reactors by deuterium activation and by the activation of lithium hydroxide.

The technician can obtain concentrated tritium samples by freezing out the water from a gas sample. This procedure may be used when titrated water vapor is present or has been formed when the hydrogen gas was passed over a catalyst. The water can be reacted with magnesium metal to liberate the hydrogen gas.

Hydrogen also can be separated from other gases if the mixture is passed through a bed of finely divided uranium metal. The uranium metal first is prepared by hydrogenation at 225 to 250°C to form uranium hydride. The hydride then is heated until it decomposes and yields a very finely powdered uranium. This material has a very good absorption coefficient for hydrogen, and the collected hydrogen can be removed for analysis simply by heating the material to 435°C. This technique produces samples of very pure hydrogen gas.

Carbon-14

Carbon-14 is a beta emitter that exhibits a maximum beta energy of 0.155 MeV and a half-life of 5600 years. Atmospheric carbon-14 usually is in the form of carbon dioxide, having been formed by neutron activation of nitrogen-14. However, gaseous hydrocarbons containing carbon-14 often are produced in laboratories, thereby yielding another source of airborne carbon-14.

Gaseous carbon-14 usually is collected as carbon dioxide, a carbonate. The filtered gas is collected when drawn through sodium or barium hydroxide to produce the appropriate carbonate. Carbon dioxide also can be collected by condensation by use of liquid nitrogen.

In either case, the carbon dioxide is liberated and purified by reprecipitation as strontium carbonate. Strontium carbonate is preferred over barium carbonate because of the radium normally associated with barium.

The strontium carbonate may be counted directly, or it may be converted to acetylene gas for counting. Still another method converts the carbon dioxide into benzene for liquid scintillation counting. All of these methods can produce accurate results.

NOBLE GASES

The neutron activation product, argon-41, and the fission products that are noble gases are important as atmospheric radioactive gases. All of these are beta emitters having comparatively short half-lives. The fission products of particular interest include xenon-133, 135, 137, 138, 139, 140, 141; and krypton-85, 89, 91, and 92.

Three methods are available for the measurement of airborne concentrations of these gases. One is by direct measurement; a second is by determining their longer-lived daughter activities; and the third is by separation based on physical phenomena, followed by counting.

Direct Counting

Direct-counting techniques primarily are employed for argon-41 counting. This radioisotope normally is produced through activation of the argon component of air dissolved in reactor coolant water and may be vented to the atmosphere.

The sample is collected by the purging of either a 100-ml or an 18-ml counting chamber. The counting chamber then is mounted to a detector for counting. The detector may be either a GM tube with appropriate instrumentation or a scintillation detector connected to a single channel adjusted to count the 1.29-MeV gamma photon. Figure 20-4-10 illustrates argon-41 collecting and counting chambers. Argon-41 has a relatively short half-life, which must be remembered when it is counted.

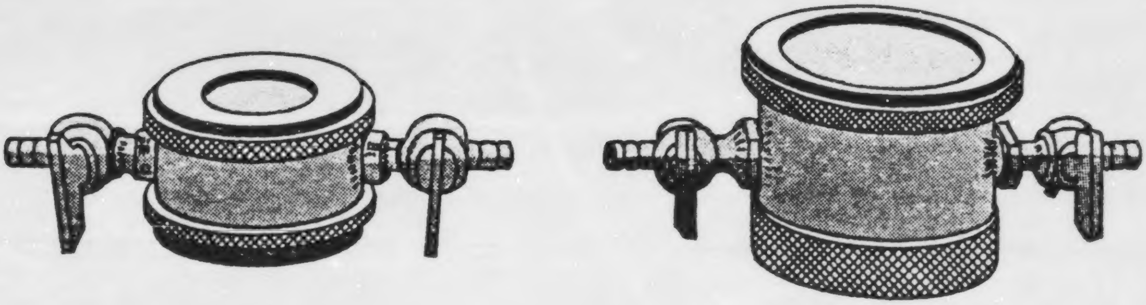


Figure 20-4-10. Argon-41 counting chambers.

Daughter Product Activities

Because of the particular nature and relatively longer half-lives of noble gas daughter products, the daughters of fission products that are noble gases are more important than the gases themselves. The methods of radiochemical separation and gamma spectroscopy are utilized in analytical procedures.

The radiochemical procedure collects the daughter products onto a stainless steel frit in a prefiltered trap. After collection, the daughters are etched from the frit and analyzed radiochemically. This technique has been shown to be efficient for these parent/daughter radioisotope combinations: ^{89}Kr/^{89}Sr, ^{137}Xe/^{137}Cs, ^{140}Xe/^{140}La, and ^{141}Xe/^{141}Ce.

Gamma spectroscopy is used to determine first- and second-generation daughters of gaseous fission products. This method permits measurement of shorter-lived components than does the radiochemical method. The samples are collected by electrostatic precipitation and then subjected to gamma spectrographic analysis. The counting techniques of time-interval counting and spectrum stripping are employed in the analysis. This technique is useful for the following parent/daughter radionuclides: ^{89}Kr/^{89}Rb, ^{91}Kr/^{91}Sr, ^{92}Kr/^{92}Sr, ^{137}Xe/^{137}Cs, ^{138}Xe/^{138}Cs, ^{139}Xe/^{139}Ba, ^{140}Xe/^{140}La.

Physical Processes

Different physical reaction rates are exhibited by different elements and by isotopes of the same element when subjected to physical phenomena such as diffusion, adsorption and absorption. Since the noble gases are almost chemically inert, physical rate processes are required for the separation of a specific gas from a mixture.

One technique that has proved useful is thermal gravitational diffusion. With this method, the gases are flowed through a chamber in which one wall is heated and the opposite wall cooled. Molecules of the lighter gases collect at the top of the heated wall, and molecules of the heavier gases collect at the bottom of the cold wall. Vertically aligned fibers of silica in the diffusion cell increase separation of neon and argon, and a structure of porous stainless steel is best for neon isotope enrichment. Helium-neon separations work best in a cell that contains no dividing structures.

Chromatographic separation of noble gases is possible because they are intermittently adsorbed and desorbed from activated charcoal, providing a delay relative to the carrier gas. Since each gas has a different adsorption coefficient, the means of chromatographic separation is provided.

Xenon can be absorbed by high-density graphite if heated to 750-1000°C. Quenching of the graphite in cold mercury seals in the absorbed xenon. The material then can be counted directly, or the xenon can be stripped out for counting.

ACTIVITY CONCENTRATION CALCULATIONS

All of the preceding is necessary in order to deterine two bits of data needed to calculate the activity concentration for comparison with MPC's. The data necessary are (1) the volume of the gaseous sample and (2) the activity contained therein. When these two factors are known, the activity per unit volume can be calculated from Equation 20-4-1.

$$\text{Activity Concentration} = \frac{\text{Total Sample Activity}}{\text{Total Sample Volume}}$$

Equation 20-4-1
(Equation = Eq.)

where:

Activity concentration is in μCi/ml.
Total sample activity is in μCi.
Total sample volume is in ml.

EXAMPLE

Given: A 100-ml sample of air is found to contain 2.7×10^{-3} μCi of ^{41}Ar.

Find: The activity concentration per ml of air.

Solution: Use Equation 20-4-1:

$$\text{Activity concentration} = \frac{\text{total sample activity}}{\text{total sample volume}}$$

$$^{41}\text{Ar activity concentration} = \frac{2.7 \times 10^{-3}\ \mu\text{Ci}}{100\ \text{ml}}$$

$$^{41}\text{Ar activity concentration} = 2.7 \times 10^{-5}\ \mu\text{Ci/ml.}$$

Since activity concentrations derived from these calculations are in the same units as those published in 10CFR20, comparison is both easy and meaningful.

EXERCISES

1. Write a procedure for the collection of a gaseous sample by adsorption.
2. Write a procedure for the collection of a gaseous sample by absorption.
3. Write a procedure for the collection of a grab sample from the atmosphere.
4. Prepare a list of gaseous radionuclides and the types of detectors with which each is counted.

MATERIALS

Adsorption column sampling apparatus.
Activated alumina.
0.3-micron membrane filters.
Timer.
Vacuum desorption flask.

Gas counting chambers, 100 and 18 ml.
Gamma integral counter.
Gamma spectrograph.
Gas absorption train.
Saturated barium hydroxide solution.
Saturated strontium chloride solution.
Vacuum filtration apparatus.
Glass fiber filter paper.
Gas generator.
Ammonium hydroxide.
Hydrochloric acid.
Beta counter.
100-ml hand aspirator.
Prefilter holder.
Scintillation detector.
Pulse height analyzer.

PROCEDURES

ADSORPTION ONTO AN ALUMINA COLUMN

1. Set up an activated alumina adsorption column according to manufacturer's instruction sheet.
2. Remove particulates with a 0.3-micron membrane prefilter.
3. Turn on sampler; adjust flow rate to manufacturer's recommended sampling rate.
4. Record in Data Table 20-4-1 the time and the flow rate when the instrument is started.
5. Sample for 1 hour.

6. Record time and air flow rate; turn the machine off.
7. Place adsorption column into heated vacuum desorption flask.
8. Following manufacturer's procedure, desorb the gases and drive them into the counting chamber.
9. Record the time and take an integrated gamma count and a gamma spectrum count.
10. Take additional integrated gamma counts as necessary to determine the half-life of the gamma ray.
11. By gamma spectroscopy, identify the gamma emitter present.
12. Record data in Data Table 20-4-1.

ABSORPTION SAMPLING OF ^{14}C

1. Set up a gas absorption train, using 4 two-liter gas absorption bottles.
2. Fill absorption bottles with a saturated solution of barium hydroxide.
3. Connect a 0.3-micron membrane prefilter to remove particulates.
4. Turn on apparatus; adjust flow rate to manufacturer's specification.
5. Record time and gas flow rate in Data Table 20-4-2.
6. Collect the sample for 30 minutes.
7. Record time and gas flow rate; turn off the apparatus.
8. Collect the barium carbonate by vacuum filtration through a glass fiber filter paper.
9. Wash residue on the filter paper one time with saturated barium hydroxide.
10. Transfer filter paper and residue to a gas generator.

11. Regenerate the carbon dioxide by adding dilute hydrochloric acid to the generator.
12. Collect the carbon dioxide by reacting it with ammonium hydroxide to form ammonium carbonate.
13. Precipitate strontium carbonate by adding saturated strontium chloride solution until precipitation is complete.
14. Collect the strontium carbonate by vacuum filtration, using a glass fiber filter paper.
15. Dry, mount, and beta count the sample.
16. Calculate the ^{14}C concentration in µCi/ml.
17. Record data in Data Table 20-4-2.

GRAB SAMPLING FOR ^{41}Ar

1. Connect a 0.3-micron membrane prefilter to a 100-ml gas counting chamber.
2. Connect a 100-ml hand aspirator to the opposite stopcock on the counting chamber.
3. Collect sample by opening both stopcock valves and squeezing aspirator bulb at least 10 times.
4. Close stopcocks; record time of collection in Data Table 20-4-3.
5. Disconnect prefilter and aspirator from counting chamber.
6. Count the sample on a scintillation detector connected to a pulse height analyzer adjusted to count the 1.29-MeV gamma photon from ^{41}Ar.
7. Calculate activity per ml of the sample.
8. Record data in Data Table 20-4-3.

150

DATA TABLE 20-4-1.
ALUMINA ADSORPTION

SAMPLE COLLECTION	TIME	GAS FLOW
Start	________	________
Stop	________	________
Total	________	________

Integrated counts	Time	Counts per Minute
	________	________________
	________	________________
	________	________________
	________	________________
	________	________________

Sample half-life ________________

Gamma spectroscopy

Isotopes present ____________________________

____

____

____

____

DATA TABLE 20-4-2.
^{14}C ABSORPTION

SAMPLE COLLECTION	TIME	GAS FLOW
Start	________	__________
Stop	________	__________
Total	________	__________
SAMPLE COUNTING	COUNTS PER MINUTE	MICROCURIES
Carbon-14 activity	_________________	___________
^{14}C µCi/ml of sample	_________________	

DATA TABLE 20-4-3.
ATMOSPHERIC GRAB SAMPLE FOR ^{41}Ar

Volume of sample counting chamber _______________ ml
Sample counts per minute _______________
Sample activity in microcuries _______________ µCi
Sample activity concentration _______________ µCi/ml

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TEST

1. Write a procedure for the collection of gaseous ^{131}I by adsorption.

2. Write a procedure for the collection of $^{14}CO_2$ by absorption.

3. Write a procedure for the grab sample collection of ^{41}Ar in the air.

4. Given a sample that produces 912 counts per minute initially and 417 counts per minute 2 hours later, calculate half-life of the sample.

5. Given a 100-ml sample of gas containing 3.6×10^{-2} µCi of ^{41}Ar, calculate the number of µCi/ml.

6. Given the following gaseous radioisotopes, list the types of detectors that may be used for each:
   a. Tritium

   b. $^{14}CO_2$

   c. ^{41}Ar

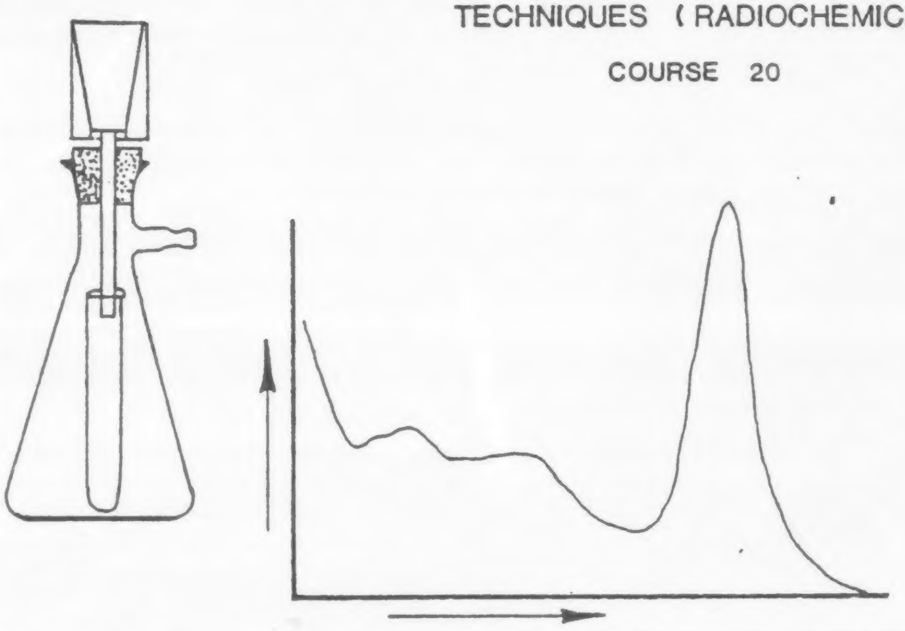
NUCLEAR TECHNOLOGY

MODULE 20-5

WATER MONITORING TECHNIQUES - DISSOLVED SOLIDS

RADIATION MONITORING
TECHNIQUES (RADIOCHEMICAL)

COURSE 20



CENTER FOR OCCUPATIONAL RESEARCH AND DEVELOPMENT

COURSE MODULES

COURSE 20 RADIATION MONITORING TECHNIQUES (RADIOCHEMICAL)

MODULE 20-1	Radiation Sample Preparation – Liquid
MODULE 20-2	Radiation Sample Preparation – Solid
MODULE 20-3	Air Monitoring Techniques – Particulate
MODULE 20-4	Air Monitoring Techniques – Gaseous
⇨ MODULE 20-5	Water Monitoring Techniques – Dissolved Solids
MODULE 20-6	Water Monitoring Techniques – Suspended Solids
MODULE 20-7	Radiation Monitoring of Biological Samples

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INTRODUCTION

The radioactive materials found in water originate from both natural and man-made sources. Natural radioactivity found in water is usually in quantities of less than a picocurie of alpha activity, although the beta activity may amount to tens of picocuries.

Base lines of radioactive contamination of water must be established, and any change in the total activity or in the various components of that total must be monitored continuously. This module is concerned with that portion of the radioactive constituent of water held in solution.

MODULE PREREQUISITES

The student should have completed a course in chemistry; Course 1, "Radiation Physics"; and Course 11, "Radiation Detection and Measurement."

OBJECTIVES

Upon completion of this module, the student should be able to:

1. Write the procedure for the collection of a liquid grab sample.
2. Write the procedure for the collection of a liquid composite sample.
3. Given a sample of water containing dissolved solids, but no suspended solids, select a procedure for the

collection of the dissolved solids and for the determination of the amount and type of radioactivity present.

4. Given a volume of water and the amount and type of radiations emitted, calculate the activity per unit volume of the solution.
5. Given a water sample, select the proper volume for analysis.

SAMPLE COLLECTION

Accurate laboratory test results indicate only the condition of the sample used to obtain those results. The sample, therefore, must be representative of the material sampled if the data will be useful.

There are three rules to be observed in sampling. Failure to strictly observe any one of the three may render the sample useless.

The first rule is in sampling cleanliness. All containers, including stoppers or caps, must be clean, as must all measuring devices, cups, graduates, etc. Anything that comes in contact with the sample must be clean to prevent contamination of the sample.

The second rule, accuracy, applies to records concerning the sample. A label should be attached to the sample container as soon as the sample is collected. The label should include the type of sample (grab or composite), source of the sample, location of sampling point, date and time of sample, temperature of sample, and possibly a note on recent weather conditions. The weather conditions may be particularly important in the case of an environmental sample collected an hour after a three-inch rain.

The third rule of sampling is refrigeration. Many samples may contain living organisms, which will continue to grow and metabolize materials in the sample unless their life processes are stopped. This is best done by storing the sample at 10°C. Such cold temperature not only stops the life processes of the living organisms, but also prevents decay of the organisms prior to analysis.

A sample, by definition, is a part of anything that is presented as evidence of the quality of the whole. Therefore, the quality must be maintained between the time of collection and the time of analysis.

There are two types of samples used in the analysis of water: These are "grab" samples and "composite" samples. Each has its use in describing conditions.

GRAB SAMPLES

A grab sample is a sample taken at a specific time and place, with no regard to volume or flow conditions. It simply provides information about concentration.

Care must be taken to ensure that the grab sample contains only the liquid being sampled. In the collection of environmental samples from a flowing stream, the sample should be taken from the middle of the stream, but must not include mud from the bottom. Figure 20-5-1 is an illustration of a grab sampler.

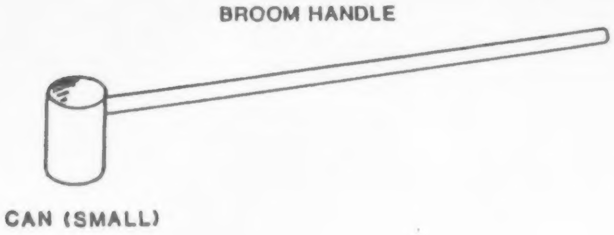

Figure 20-5-1. Typical grab sampler.

Two liters usually is considered to be the minimum volume of a sample to be submitted for analysis. This volume may be reduced considerably for some specific examinations, but it is much better to have too much sample than not enough. Considering that the volume of a sample is likely to be such a small part of the total volume for comparison, most workers collect a four-liter sample to provide a more representative sample and to ensure sufficient volume for retesting if required.

COMPOSITE SAMPLES

The second type of sample is the composite sample. A composite sample is one taken in several parts over a specified period of time and then combined to form a representative sample. In other words, a composite sample is a series of grab samples combined to make one sample. The procedure may be done manually or by machine. Figure 20-5-2 depicts an automatic composite sampler. Composite samples minimize variations and give a more average representation of conditions.

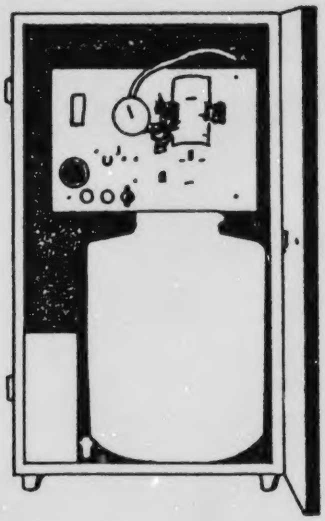

Figure 20-5-2. An automatic composite sampler.

A variation of the composite sample is the weighted composite sample. In this type of sample, the volume of each grab is made proportional to the flow rate: The higher the flow, the larger the portion taken at that time. Thus, the final composite sample is representative of the total volume that has flowed past the sampling point. This type of sample provides the best information about a continuously flowing stream.

The volume of each grab required to produce a weighted composite sample can be calculated from Equation 20-5-1.

$$\text{Volume per sample} = \frac{\text{Total sample volume}}{\text{No. of samples}} \times \frac{\text{Flow rate at sample time}}{\text{Average flow rate}}$$

Equation 20-5-1
(Equation = Eq.)

The number of samples is equal to the total time of sampling, divided by the time interval between sampling, plus one.

EXAMPLE

Given: A power plant has a cooling water discharge average of 500 gallons per minute. The discharge is to be sampled every half hour for twenty-four hours. At one particular sample time, the discharge flow was found to be 700 gallons per minute. The final composite sample volume should be 4 liters.

Find: The volume of the grab at this flow rate.

Solution: Using Equation 20-5-1 –

$$\text{Volume} = \frac{\text{Total sample volume}}{\text{No. of sample}} \times \frac{\text{Flow rate at sample time}}{\text{Average flow rate}}$$

$$= \frac{4000\ \text{ml}}{24\ \text{hr}/0.5\ \text{hr} + 1} \times \frac{700\ \text{gpm}}{500\ \text{gpm}}$$

$$\text{Volume} = 114.29\ \text{ml} = 114\ \text{ml}.$$

When a sample is being collected, both the collecting container and the storage/transport container should be rinsed three times with the solution of the sample. Rinsing will help to ensure that all extraneous contaminants have been removed. Additionally, it is a good idea for the technician to discuss special sampling requirements with the laboratory personnel who are to do the testing.

Samples that are to be analyzed for the presence of metals should be acidified by the addition of 2 ml of concentrated nitric acid per liter of sample. The acid helps to prevent adsorbence to the glass or to the plastic of the container.

ADDITIONAL PROCEDURES

Glass or polyethylene bottles are considered to be appropriate vessels for the collection of samples for analysis. Carriers or preservatives should not be added to samples that are to be examined for suspended or dissolved materials. This means that there should be as little delay as possible between collection and analysis of the sample. Changes in the distribution of activity between water, solids, and the container surface will occur during storage; and the short-lived radionuclides will decay.

SAMPLE PREPARATION

Preparation of a sample normally is a part of the analytical procedure. The exception is in gamma spectroscopy of a water sample. This procedure usually is nondestructive and simply involves the pouring of a 3.5-liter sample of clear water into a beaker, where it is counted. The sample then may be subjected to further analytical procedures. However, if the suspended particles have a tendency to settle, agar-agar with sequestering chemicals may be added to prevent settling while the sample is counted. Usually, gamma spectrometry can be improved for nonvolatile radionuclides by evaporation of the sample to a small volume or to dryness in order to improve the counting geometry.

Clear waters require no additional preparation. However, if a sample for the analysis of radiostrontium is to be stored for more than 24 hours, the sample should be pretreated with strontium carrier (as prescribed in the analytical procedure) and then preserved by acidification to a pH of 4.0.

As a general rule, the use of carriers in the analytical procedure is ineffective unless any solids present are converted to a soluble phase. This is true, although the solids may interfere with the radioanalytical procedure. Table 20-5-1 indicates the usual distribution of the common radioelements between solution and suspension.

TABLE 20-5-1.
USUAL DISTRIBUTION OF COMMON RADIOELEMENTS BETWEEN SOLID AND LIQUID PHASES IN INDUSTRIAL EFFLUENTS.

Solution	Suspension
HCO_3	Ce
Co	Cs
Cr	Mn
Cs	Nb
H	P
I	Pm
K	Pu
Ra	Ra
Rn	Sc
Ru	Th
Sb	U
Sr	Y
	Zn
	Zr

Additionally, these radioelements may exhibit unusual chemical characteristics because of the presence of various complexing agents or their source of discharge. For example, radioiodine from a hospital complex probably will be a mixture of iodides and organic compounds, with different characteristics from the elemental form employed in fission-product processing.

EVAPORATION

Evaporative sample preparation techniques are utilized to concentrate large volumes of sample and for gross alpha and gross beta activity determination of dissolved solids in clean water. Evaporative concentration should be performed on a hot plate or a steam bath. Care must be taken to prevent the solution from boiling, as this may cause loss of some of the sample. This procedure should not be used if volatile material is present unless compensation is made for its loss.

Evaporative sample preparation for gross alpha and gross beta determination requires certain special precautions. Care must be taken to ensure that the residue will not create sample self-absorption problems. The final counting sample must not contain more than 5 mg of residue per square centimeter for alpha counting and not more than 10 mg per square centimeter for beta counting. The proper volume for evaporation should be selected by measurement of the sample specific conductance.

After the appropriate sample volume has been determined, either of two techniques for evaporation may be used. The first method is very direct: The sample is added directly, in small increments, to a tared counting planchet and is evaporated while the planchet is maintained just below boiling temperature. After the sample is evaporated, the planchet is placed in a drying oven at 105°C to complete the drying and is cooled in a desiccator. The sample then is ready for counting.

The second method is to place the correct volume of sample in a pyrex beaker or evaporating dish and to acidify to a pH of 4 to 6 with either hydrochloric acid or nitric acid. The sample is evaporated on a hot plate or in a steam bath to near dryness. The solids must not be baked onto the evaporating vessel. The solution is transferred quantitatively to a tared planchet and the drying completed in a drying oven at 105°C. The sample should be cooled in a desiccator before it is counted. NOTE: Excess alkalinity or mineral acidity is corrosive to alumnium planchets.

RADIOCHEMICAL METHODS

The most common radiochemical analyses for dissolved solids are for radiostrontium, radiobarium, radium, and tritium. Each of these will be discussed briefly, and specific procedures will be presented in the Procedures section of this module.

Radiostrontium

Dissolved strontium is removed from water by the oxalate precipitation method. The strontium then is precipitated as strontium nitrate in concentrated nitric acid; and calcium, barium, radium, and the rare earths are separated as soluble nitrates. Yttrium-90 is allowed to grow from 7 to 14 days and then separated from the strontium. The yttrium-90 is measured to estimate the strontium-90, and the combined strontium-89 and -90 activity is measured in order that the strontium-89 can be estimated by difference. However, if the water sample is ocean water or is a saline water, radiostrontium is determined by the basic carbonate method. This procedure also may be used for fresh water.

When radiostrontium is determined by the basic carbonate method, magnesium is removed by precipitation with 8-hydroxyquinoline. The strontium is precipitated as the carbonate, converted to the nitrate, and purified by hydroxide scavenging. Strontium then is reprecipitated as the carbonate and counted.

Radiobarium

Barium-140 is one of the more abundant radionuclides found in mixed fission products. It can be determined by gamma counting of the 1.60-MeV gamma photon of its daughter, lanthanum-140. Alternatively, radiobarium can be chemically isolated from other radionuclides and beta counted.

Radiobarium usually is determined by the basic carbonate method since this procedure is compatible with both the oxalate precipitation method and the tributyl phosphate extraction methods for radiostrontium.

Strontium, barium, and the alkaline earths are precipitated as carbonates. The precipitate then is dissolved in dilute nitric acid and reprecipitated from concentrated nitric acid. The precipitate is dissolved in water, and the rare earths are precipitated as hydroxides. Barium and radium chromates are precipitated from the supernate, and the barium is separated from the radium. The barium-140 then may be beta counted and allowed to come into equilibrium with its daughter, lanthanum-140, and gamma counted.

Radium

Since radium-226 is one of the more important of the radionuclides that contribute to internal radiation exposure, any time the gross alpha activity in potable water exceeds 3 picocuries per liter, the sample should be analyzed for radium. There are two principal methods in current use. The first involves precipitation and purification of radium, with barium as the sulfate, followed by the alpha counting of the precipitate. This method is useful as a screening technique, but does not differentiate between the isotopes of radium.

When the total radium content exceeds the drinking water standard, a method specific for radium-220 is required. This method is based upon the concentration of radium, collection of the radon-222 emanation, and alpha counting of the radon-222. This method is specific for radium-226.

Tritium

The tritium content of water is determined by liquid scintillation counting. The sample first is purified by distillation, then is mixed with the scintillation cocktail for counting. Close attention to this procedure will produce a minimum level of detectability of approximately 1.20 nanocurices per liter of water.

COUNTING TECHNIQUES

ALPHA ACTIVITY

Alpha activities probably are best counted by an internal proportional counter. However, some excellent alpha scintillation counters are on the market, and gaseous alpha emitters are counted very well by scintillation techniques.

Solid samples, counted for alpha activity, must be prepared to eliminate sample self-absorption as much as possible. Samples for internal proportional counting must have a density thickness of no more than 5 mg per square centimeter, and samples for scintillation counting must have a thickness of no more than 3 mg per square centimeter. This difference in maximum acceptable thickness is due to air absorption in the scintillation counters. The exception occurs when the scintillation counting chamber is evacuated.

BETA ACTIVITY

Beta activities can be counted effectively by a variety of methods. Proportional counting is very effective. Samples for internal proportional counting must be prepared in such a manner as to ensure good adherence of the residue to the planchet. However, if a thin-window proportional counter is used, sample adherence is not so critical, although there is an efficiency loss.

End-window GM counters are effective, but there is some loss of weak betas. Most alpha activity is filtered out by the window thickness and the air gap between the window and the sample. Planchet diameter must be less than that of the detector.

Weak and very weak beta emitters are best counted in a liquid scintillation counter. Counting efficiencies can approach 100% because absorption losses are eliminated. Care must be taken during sample preparation to ensure that the sample is completely dehydrated or that the scintillation cocktail is mixable with water.

GAMMA COUNTING

Sample preparation for gamma counting is not quite so critical. This is due to the penetrating abilities of the gamma radiations; however, gaseous detectors are not very efficient for gamma rays. The efficiency of end-window GM detectors may be increased somewhat if a thin lead absorber is placed in front of the window to allow counting of photoelectrons knocked out of the absorber by the photoelectric absorption of gamma rays. However, this procedure is somewhat "arty."

Probably one of the more efficient methods is scintillation counting with a thallium-activated sodium iodide crystal. Efficiencies of these systems are usually less than 50%.

Solid-state detectors are quite in vogue at the present time, although their efficiency is considerably less than that of soidum iodide crystals. They are especially useful in gamma spectroscopy since their resolution is far superior to other scintillation systems.

Two considerations must be given to a sample being prepared for gamma counting, regardless of whether it is a liquid sample for well counting or a solid sample for counting with a standard detector. If the sample is an alpha or beta emitter, as well as a gamma emitter, provision must be made to prevent the alpha or beta particles from entering the detector. These radiations also can produce scintillations and can change the count rate. Also, any absorber placed between the source and the detector to prevent these particles from reaching the detector should be a low-Z material to reduce production of bremsstrahlung radiation that would be counted as gamma rays.

SPECIFIC PROCEDURES

The following specific procedures and techniques are standard. They may be used, with variations, for other elements than those specifically mentioned.

GROSS ALPHA AND BETA ACTIVITY

Care must be taken to limit sample self-absorption in the preparation of samples for the determination of the gross alpha and beta activity of dissolved solids. Samples for alpha assay are limited to a final density thickness of 5 mg/cm^2, and samples for beta assay are limited to no more than 10 mg/cm^2. Additionally, sample residues that have a tendency for portions to become airborne should be treated with a few drops of Lucite solution to act as a binder. This treatment helps to prevent loss of sample and contamination of the counting system.

Procedure:

1. Filter sample through a glass-fiber filter to remove any suspended matter.
2. Place a conductivity cell in filtered sample; measure conductivity of the solution.
3. From Equation 20-5-2, calculate the approximate dissolved solids content in mg/l as calcium carbonate.

$$CaCO_3 \text{ mg/l} = \mu\text{mho conductivity} \times 0.5 \text{ mg/l/}\mu\text{mho} \qquad \text{Eq. 20-5-2}$$

EXAMPLE

Given: A solution that has a conductivity of 5000 µmho.

Find: Approximate dissolved solids content in mg/l as $CaCO_3$.

Solution: $CaCO_3$ mg/l = µmho x 0.5 mg/l/µmho

= 5000 µmho x 0.5 mg/l/µmho

$CaCO_3$ mg/l = 2500 mg/l.

4. Calculate area of planchet to be used.
5. Calculate volume of sample required to produce a deposit of 5 mg/cm^2 for alpha assay and 10 mg/cm^2 for beta assay.

EXAMPLE

Given: Sample containing 2500 mg/l of dissolved solids as $CaCO_3$ and planchet with area of 20 cm^2.

Find: Volume of sample required for alpha assay.

Solution:

$$\text{Amount of solids permitted} = 5\ \text{mg/cm}^2 \times 20\ \text{cm}^2$$

$$\text{Amount of solids permitted} = 100\ \text{mg.}$$

$$\text{Volume of sample} = \frac{1000\ \text{ml}}{\text{CaCO}_3\ \text{mg/ml}} \times \text{Amount of solids permitted}$$

$$= \frac{1000\ \text{ml}}{2500\ \text{mg/ml}} \times 100\ \text{mg}$$

$$\text{Volume of sample} = 40\ \text{ml.}$$

6. Measure required volume of sample into a clean beaker.
7. Evaporate sample in beaker to near dryness on a hot plate. Be careful not to boil sample or to bake residue.
8. Transfer concentrate to tared metal planchet.
9. Rinse walls of the beaker with a few drops of 3N nitric acid, and combine with concentrate in metal planchet.
10. Dry sample in a drying oven at 103-105°C.
11. Cool in a desiccator; weigh.
12. Repeat Steps 10 and 11 until weight is constant.
13. If residue has a tendency to flake, treat it with a few drops of Lucite solution, and dry to a constant weight.
14 Store sample in a desiccator until it is to be counted.
15. Count gross alpha and gross beta activities. This probably is best accomplished with an internal proportional counter, although other methods can be used.
16. Calculate activities according to Equations 20-5-3 and 20-5-4.

$$\text{Gross Alpha Activity, pCi/l} = \frac{C_a}{V \times E \times A} \qquad \text{Eq. 20-5-3}$$

where:

C_a = net alpha count rate in cpm.
V = volume of sample in liters.
E = efficiency, for counting alpha particles from an infinitely thin sample, in cpm/Ci.
A = sample self-absorption correction factor.

$$\text{Gross Beta Activity, pCi/l} = \frac{C_b}{V \times E_b \times A_b} \qquad \text{Eq. 20-5-4}$$

where:

C_b = net beta count rate in cpm.
V = volume of sample in liters.
E_b = gross beta counting efficiency in cpm/pCi.
A_b = self-absorption factor for beta counting.

NOTE: If alpha sample thickness exceeds 5 mg/cm^2 or beta sample thickness exceeds 10 mg/cm^2, infinite thickness must be assumed. Activities are reported as approximate, or examinations must be repeated with a smaller sample. If ^{90}Sr/^{90}Y self-absorption factors are used, results are reported as beta-gamma activity, not as gross activity.

EXAMPLE

Given: A sample has a net alpha count rate of 34.8 cpm and a net beta count rate of 97.2 cpm. The sample thickness is 4 mg/cm^2, and it was prepared from a volume of 150 ml. Alpha counting efficiency is 1.06 cpm/pCi; beta efficiency is 0.97 cpm/pCi; and self-absorption factors for alpha and beta are 0.98 and 0.79, respectively.

Find: Gross alpha and gross beta activities.

Solution:

Gross alpha activity, from Equation 20-5-3:

$$\text{Gross alpha activity, pCi/l} = \frac{C_a}{V \times E \times A}$$

$$= \frac{34.8 \text{ cpm}}{0.15 \text{ l} \times 1.06 \text{ cpm/pCi} \times 0.98}$$

Gross alpha activity = 223 pCi/l.

Gross beta activity, from Equation 20-5-4:

$$\text{Gross beta activity} = \frac{C_b}{V \times E_b \times A_b}$$

$$= \frac{97.2 \text{ cpm}}{0.15 \text{ l} \times 0.97 \text{ cpm/pCi} \times 0.79}$$

Gross beta activity = 846 pCi/l.

STRONTIUM-89 and -90

Radiostrontium is one of the most important of the fission products that contribute to the body burden of beta emitters. As the strontium body burden comes principally from food and drinking water, it is important to assess the radiostrontium content of these pathways.

Procedure:

1. Prepare 25 ml each of the following carrier solutions:
   Barium, $Ba(NO_3)_2$, 13.32 mg/ml.

Calcium, $Ca(NO_3)_2 \cdot 4H_2O$, 235.68 mg/ml.
Strontium, $Sr(NO_3)_2$, 48.31 mg/ml.
Yttrium, $Y(NO_3)_3 \cdot H_2O$.

2. Filter sample to remove suspended matter.
3. Place 1 liter of filtrate into a 2-liter beaker; acidify with 2 ml of concentrated nitric acid.
4. Add 1 ml each of strontium and barium carrier solutions.
5. Add 10 ml of calcium carrier solution.
6. Stir thoroughly.
7. Add 125 ml of saturated oxalic acid; continue to stir for 5 minutes.
8. Using a pH meter to measure change, add 15N ammonium hydroxide with continuous stirring until the solution is at a pH of 3.0.
9. Allow solution to stand for 6 hours or overnight.
10. Filter material through a medium-porosity glass-fiber filter, using suction; discard filtrate.
11. Rinse precipitate in beaker and that on filter with 0.5% ammonium oxalate 3 times.
12. Wash filter residue back into original beaker, using 5 10-ml portions each of hot, concentrated nitric acid. Rinse down the sides of beaker with 5 5-ml portions of hot, concentrated nitric acid.
13. Evaporate solution to near dryness; add 50 ml of concentrated nitric acid; heat.
14. Repeat Step 13 until residue is colorless.
15. Quantitatively transfer residue to a 50-ml centrifuge.
16. Cool in a refrigerator overnight; centrifuge at 1500 to 1800 rpm for 10 minutes.
17. Decant, discarding supernate.
18. Dissolve precipitate in 5 ml of 3N HNO_3; add 10 ml of fuming nitric acid.

19. Centrifuge and decant, discarding decantate.
20. Dissolve precipitate in 5 ml of distilled water; add 3 drops of bromcresol green indicator.
21. Titrate with 6N ammonium hydroxide until color changes from yellow to blue.
22. Back titrate with 0.5N nitric acid until solution just turns yellow.
23. Add 5 ml of ammonium acetate buffer (pH 5); heat in a boiling-water bath for 5 minutes.
24. Add 1 ml of 0.25N Na_2CrO_4; heat in water bath for 15 minutes.
25. Cool to room temperature; filter through a Whatman No. 42 filter paper, saving both precipitate and filtrate.
26. Wash precipitate 3 times with 5-ml portions of warm water, adding washings to filtrate.
27. Save precipitate for the analysis of radiobarium.
28. Prepare 10 ml of a scavenger solution that contains ferric chloride, 58 mg/ml; cerous nitrate, 31 mg/ml; and zirconyl chloride, 35 mg/ml.
29. Add 5 drops of scavenger solution to filtrate of Step 26.
30. Add 2 drops of 12N HCl and 5 drops of 30% H_2O_2.
31. Place in boiling-water bath for 2 minutes; add 15N ammonium hydroxide with constant stirring until a precipitate forms.
32. Filter through a Whatman No. 42 filter paper, saving filtrate.
33. Wash residue on the filter paper 3 times with 5-ml portions of 6N ammonium hydroxide, adding washings to the filtrate. Discard residue and filter paper.
34. Evaporate filtrate to a volume of approximately 10 ml. Quantitatively transfer filtrate to a 50-ml centrifuge tube, using 2 5-ml portions of 15N ammonium hydroxide to rinse solution into centrifuge tube.

35. Add 2 ml of 3N Na_2CO_3; mix thoroughly; cool; and centrifuge.

36. Check for completeness of precipitation by adding 2 drops of sodium carbonate solution to clear supernate. If precipitation is not complete, add 5 ml of 15N ammonium hydroxide; and repeat Steps 35 and 36.

37. When precipitation is complete, decant and discard supernate.

38. Dissolve precipitate in 4 ml of 3N nitric acid.
NOTE: It may be necessary to heat for 2 or 3 minutes in boiling-water bath.

39. Add 10 ml of fuming nitric acid; cool with running water; and centrifuge. Decant and discard supernate, saving precipitate.

40. Repeat Steps 38 and 39. RECORD DATE AND TIME AS THE BEGINNING OF YTTRIUM-90 REGROWTH.

41. Add 10 ml of water, 5 ml of 15N ammonium hydroxide, and 2 ml of 3N Na_2CO_3. Stir, cool, centrifuge, and decant, discarding decantate.

42. Quantitatively transfer strontium carbonate precipitate into a glass-fiber filter, using 15N ammonium hydroxide and suction filtration.

43. Wash precipitate on the filter paper 3 times with 5-ml portions of 15N ammonium hydroxide.

44. Dry with suction and a heat lamp.

45. Count sample in a low-background beta counter; record time and count rate.

46. Dissolve radiostrontium from filter mount by rinsing filter with about 4 ml of 6N nitric acid.

47. Add 1 ml of yttirum carrier; store for 7 to 14 days.

48. Add 15N ammonium hydroxide to bromcresol green endpoint. Add a 5-ml excess of 15N ammonium hydroxide.

49. Centrifuge and decant, saving supernate. RECORD TIME AS END OF YTTRIUM-90 REGROWTH AND BEGINNING OF ITS DECAY.

50. Wash precipitate 2 times with 20-ml portions of hot water, combining and saving washings for determination of strontium carrier recovery.

51. Dissolve precipitate with 3N nitric acid.

52. Repeate Steps 42 through 44.

53. Dissolve precipitate in 5 drops of 3N nitric acid; add 25 ml of water; and heat in a 90°C water bath for 20 minutes.

54. Add saturated oxalic acid, dropwise, with constant stirring until 20 drops have been added.

55. Adjust to a pH of 2 to 3 by adding 15N ammonium hydroxide, with constant stirring.

56. Heat in 90°C water bath for 5 minutes; cool in an ice bath for 10 minutes, stirring for 5 seconds every minute.

57. Filter precipitate onto a tared glass-fiber filter paper. Use gravity filtration first; then apply suction.

58. Wash precipitate once with hot water, 3 times with 95% ethanol, and 3 times with diethyl ether.

59. Dry precipitate with suction for 2 minutes; then weigh and mount the sample for counting.

60. Count beta activity of the ^{90}Y in a low-background beta counter.

61. Calculate ^{90}Sr activity according to Equation 20-5-5.

$$\underset{\text{(pCi/l)}}{^{90}\text{Sr activity}} = \frac{C_Y}{R \times E_Y \times V \times D \times Q} \qquad \text{Eq. 20-5-5}$$

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where:

C_y = beta count rate of ^{90}Y, in cpm.

R = recorvery of strontium carrier.

E_y = efficiency for counting ^{90}Y oxalate (cpm/pCi).

V = volume of water sample.

D = decay factor $e^{-\lambda t}$ for ^{90}Y decay, where t is time from Step 49 to Step 60.

Q = saturation factor, $1-e^{-\lambda t}$, where t is time from start of regrowth in Step 40 to separation of yttrium from strontium in Step 49.

62. Calculate ^{89}Sr activity according to Equation 20-5-6.

$$^{89}Sr(pCi/l) = \frac{1}{E_r \times C}\left[\frac{C_t}{R \times V} - F[(G \times H) + (I \times J)]\right] \quad \text{Eq. 20-5-6}$$

where:

C_t = net beta count rate for total radiostrontium in cpm.

E_r = counting efficiency for ^{89}Sr as the carbonate in cpm/pCi.

C = decay factor $e^{-\lambda t}$ for ^{89}Sr where t is time from sample collection until counting (Step 45).

C_t = net beta count rate for total strontium in cpm (Step 45).

R = recovery of strontium carrier.

V = sample volume in liters.

F = strontium-90 activity in pCi/l.

G = self-absorption factor for counting radiostrontium as strontium carbonate.

H = counter efficiency for counting ^{90}Sr as the carbonate in cpm/pCi.

I = counter efficiency for counting ^{90}Y as the oxalate in cpm/pCi.

J = saturation factor $1-e^{-\lambda t}$ for the equilibrium obtained during ^{90}Y regrowth from Step 40 until counting.

NOTE: Strontium carrier recovery is determined by either flame photometry or atomic absorption techniques to examine the washings from Step 50.

RADIOSTRONTIUM IN SALINE WATER

The basic carbonate method is the procedure of choice when the sample is known to be of saline water or if it is known to contain large amounts of magnesium.

Procedure:

1. Place a 1-liter sample of clear, filtered water into a 2-liter beaker.
2. Add 1 ml of a strontium nitrate solution that contains 212.54 mg of $Sr(NO_3)$ per ml.
3. Heat to near boiling; add 10 ml of 6N ammonium hydroxide.
4. With continuous stirring, add 100 ml of a 7.5%-by-volume solution of 8-hydroxyquinoline in methanol; allow to precipitate for 20 minutes.
5. Decant and filter with suction through a double layer of Whatman No. 42 filter paper.
6. Wash precipitate 3 times with 50-ml portions of water; discard precipitate.
7. Transfer filtrate to a clean 2-liter beaker; add 30 ml of 6N sodium hydroxide; heat to near boiling.
8. With continuous stirring, slowly add 50 ml of 3N sodium carbonate.

9. Allow precipitate to settle for 20 minutes; decant and discard most of supernate.
10. Quantitatively transfer precipitate and remaining supernate to a 250-ml centrifuge flask; centrifuge for 10 minutes. Decant, discarding decantate.
11. Wash precipitate with 100 ml of cold water; centrifuge and decant, discarding washings.
12. Dry precipitate in the centrifuge flask by placing flask in a drying oven overnight at 105°C.
13. Cool centrifuge flask to room temperature, and break up precipitate cake in the flask with a stirring rod.
14. Slowly, with continuous stirring, add 30 ml of 16N nitric acid.
15. Place centrifuge flask into a 90°C water bath for 10 minutes, stirring slightly every 2 or 3 minutes. This will aid in development of a precipitate.
16. Cool for 15 minutes in an ice bath.
17. Centrifuge and decant, discarding decantate.
18. Repeat Steps 14 through 17.
19. Dissolve precipitate in 5 ml of distilled water; transfer to a 50-ml centrifuge tube.
20. Rinse centrifuge flask 2 times with 5-ml portions of water, adding washings to solution in the 50-ml centrifuge tube.
21. Add 1 ml of scavenger solution; make solution just alakline with 15N ammonium hydroxide. Digest in a 90°C water bath for 15 minutes.
22. Centrifuge; decant into a clean 50-ml centrifuge tube.
23. Repeat Steps 21 and 22, combining decantates.
24. To decantate from Step 22, add 3N sodium carbonate until precipitation of strontium carbonate is complete. Check for completeness of precipitation by centrifuging

for 1 minute and adding a drop of sodium carbonate solution.

25. When precipitation is complete, stir solution to suspend precipitate. Carefully evaporate solution to a volume of less than 10 ml, and quantitatively transfer suspension to a counting planchet. Use small portions of 0.1N nitric acid to aid in the transfer of the material. Evaporate to dryness with an infrared lamp.
26. Count total radiostrontium activity in a low-background beta counter.
27. Calculate total radiostrontium activity according to Equation 20-5-7.

$$\underset{\text{pCi/l}}{\text{Total radiostrontium}} = \frac{C_b}{E \times V \times A} \qquad \text{Eq. 20-5-7}$$

where:

C_b = net beta count rate of sample in cpm.
E = efficiency for counting total radiostrontium as the carbonate in cpm/pCi.
V = sample volume in liters.
A = radiostrontium self-absorption coefficient.

RADIOBARIUM

Barium-140 is one of the more abundant fission products. It may be determined by gamma counting of the 1.60-MeV gamma photon from its daughter lanthanum-140, or the barium can be isolated chemically and beta counted.

Procedure:

1. Follow procedure for strontium-89 and -90 (the oxalate precipitation method) through Step 27.
2. Add 5 ml of 12N hydrochloric acid to precipitate. Heat in a boiling-water bath until yellow precipitate of barium chloride remains. Solution should turn green due to the presence of the chromic ion.
3. Cool for 5 minutes in an ice bath; add 20 ml of a solution of 5:1 v/v of hydrochloric acid and diethyl ether. Stir, centrifuge, and decant, discarding supernate. If remaining precipitate is not white, repeat this Step. RECORD TIME OF FINAL DECANTATION AS BEGINNING OF ^{140}La REGROWTH.
4. Weigh a stainless steel planchet and a Whatman No. 542 filter paper, and set up a vacuum filtration apparatus.
5. Quantitatively transfer precipitate to the filter, using several 10-ml portions of cold ABSOLUTE ethyl alcohol. Use at least 40 ml of absolute ethyl alcohol.
6. Transfer filter paper with precipitate to tared planchet; dry in an oven at 105°C for at least one hour. Cool in a desiccator; weigh sample and planchet.
7. Calculate fractional recovery of the barium carrier as barium chloride; count beta activity in a low-background beta counter.
8. Calculate ^{140}Ba activity in pCi/liter according to Equation 20-5-8.

$$^{140}\text{Ba(pCi/l)} = \frac{C_b}{(V \times R \times D \times E_b) + (1.15 \times E_1 \times S)}$$

Eq. 20-5-8

where:

C_b = net count rate in cpm.

V = volume of sample in liters.

R = barium recovery fraction.

D = decay correction factor $e^{-\lambda t}$ for ^{140}Ba decay, where t is time in hours from collection of sample until time of counting.

E_b = counting efficiency for ^{140}Ba as the chloride in cpm/pCi.

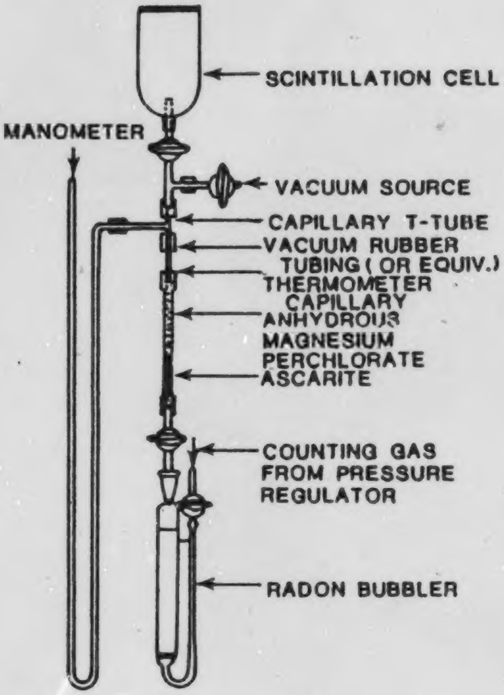
E_1 = counting efficiency for ^{140}La in the chloride precipitate in cpm/pCi.

S = correction factor $1-e^{-.015t}$ for degree of saturation from separation of barium (Step 3) until counting.

RADIUM-226

The radium-226 content of dissolved solids is determined indirectly by measurement of acitivity of its daughter, radon-222. Figure 20-5-3 depicts a radon de-emanation assembly.

Figure 20-5-3. A radon de-emanation assembly.


SCINTILLATION CELL
MANOMETER
VACUUM SOURCE
CAPILLARY T-TUBE
VACUUM RUBBER TUBING (OR EQUIV.)
THERMOMETER CAPILLARY
ANHYDROUS MAGNESIUM PERCHLORATE
ASCARITE
COUNTING GAS FROM PRESSURE REGULATOR
RADON BUBBLER

Procedure:

1. Place 250 ml of a clear, filtered water sample into a 400-ml beaker; add 2 ml of 50% ammonium sulfate, 20 ml of 10% EDTA, 1 ml of barium carrier, and 0.02 μCi of barium-133.
2. Adjust pH to 4.6; stir for 5 minutes with a magnetic stirrer.
3. Allow precipitate to settle for 20 minutes; decant, discarding decantate.
4. Quantitatively transfer precipitate to a 50-ml centrifuge tube, adding distilled water to the 50-ml mark.
5. Centrifuge for 10 minutes; decant, discarding decantate.

6. Add 30 ml of 3N sodium carbonate, stir, and place into a boiling-water bath for 30 minutes. Stir solution every 5 minutes while heating.
7. Allow to air cool to room temperature; centrifuge; and decant, discarding decantate.
8. Add 2 ml of 16N nitric acid; stir for 1 minute. Add 5 ml of water.
9. If precipitate is not complete dissolved, centrifuge and decant into a plastic counting vial that will hold approximately 30 ml. Repeat Steps 6 through 9.
10. When precipitate is completely dissolved, quantitatively transfer solution to plastic counting vial and dilute with water to 25 ml.
11. Count barium-133 with a gamma scintillation counter; calculate recovery of barium by dividing activity of the acid solution by the activity of 25 ml of water that contains 0.02 μCi of ^{133}Ba.
12. Transfer solution to a radon bubbler; remove radon-222 emanation by drawing air through the bubbler slowly for 2 minutes.
13. Close lower stopcock; allow vacuum pump to draw a partial vacuum for 3 minutes.
14. Close upper stopcock; RECORD DATE AND TIME.
15. Store bubbler containing solution for 2 to 4 weeks to allow radon-222 regrowth.
16. After 2 to 4 weeks, attach an absorption tube that contains Ascarite in the lower half and magnesium perchlorate in the upper half. Attach a radon scintillation cell to the absorption tube. Attach a helium supply to the lower stopcock of the bubbler.
17. Open stopcocks in the upper portion of the system, leaving the upper stopcock of the bubbler closed.

18. Evacuate system for 5 minutes; close stopcock to the vacuum pump, leaving the remaining stopcocks open.
19. Open upper bubbler stopcock; immediately, but slowly, begin to open lower stopcock and to allow helium to be drawn through the bubbler into the scintillation cell until system reaches equilibrium.
20. Close stopcock of the scintillation cell; RECORD DATE AND TIME. This is the beginning of radon-222 decay and of ingrowth of the daughters of radon-222.
21. Store scintillation cell for 2 hours; measure alpha activity of the cell with a scintillation counter. RECORD DATE AND TIME OF COUNTING.
22. Calculate radium-226 activity from Equation 20-5-9.

$$^{226}\text{Ra (pCi/l)} = \frac{C_a}{V \times E \times D \times R \times G} \qquad \text{Eq. 20-5-9}$$

where:

C_a = net alpha count rate in cpm.

V = sample volume in liters.

E = efficiency of de-emanation and counting. (Note: the value should be approximately 0.8.)

D = saturation factor $1-e^{-\lambda t}$, for radon-222 growth where t is the time from first radon-222 removal in Step 12 to final radon removal in Step 20.

R = recovery of barium carrier.

G = correction for radon's daughter in growth. $1-e^{-\lambda t}$, where t is time from end of second removal in Step 20 until counting.

TRITIUM

Tritium is determined by counting a sample of pure water with liquid scintillation. The water is purified by distillation.

Procedure:

1. Distill 10 to 50 ml of a filtered, clear, water sample just to dryness; BUT DO NOT BAKE RESIDUE. Collect distillate in a clean, dry container.
2. Place 13.0 ml of counting cocktail into a polyethylene counting vial.
3. Add 3.0 ml of distillate to the vial; stopper and mix content with a gentle swirling motion. Label TOP of the vial cap; place vial into a dark container.
4. Prepare a counting standard by following Steps 2 and 3: Use a titrated water standard.
5. Prepare a background standard by following Steps 2 and 3: Use a triple-distilled deep-sea water or deep-well water.
6. Place sample and standards in freezer section of a liquid scintillation counter for at least 30 minutes before counting. Count each vial for 2 to 4 hours, depending upon sample activity and instrument sensitivity.
7. Calculate tritium activity according to Equation 20-5-10.

$$^{3}H\ (pCi/l) = \frac{C_t \times 1000}{V \times E_t} \qquad \text{Eq. 20-5-10}$$

222

where:

C_t = net count rate of tritium in cpm.
V = sample volume in ml.
E_t = efficiency for counting tritium in cpm/pCi.

EXERCISES

1. Prepare a step-by-step outline of the procdure for the collection of a grab sample from a flowing stream.
2. Write a procedure for the collection of a 24-hour composite sample from a plant discharge if the discharge rate is 50 gallons per minute and the discharge is known to increase 15 gal/minute every hour for the first 5 hours; to maintain its flow rate for 2 hours; then to decrease at the rate of 5 gal/minute every hour for the remainder of the 24 hours.
3. If gross alpha and gross beta counts indicated that a water sample contained only an alpha emitter, which of the analytical procedures in this module would be applicable?
4. Define "activity per unit volume." How does it apply to dissolved solids?
5. Why would it probably be unwise to evaporate a 1-liter sample for gross alpha and gross beta examination?

MATERIALS

Ammonium hydroxide.
Ammonium sulfate.
Ascarite, 20-30 mesh.
Barium-133 tracer.
Barium nitrate.
Disodium EDTA.
Magnesium perchlorate.
Nitric acid.
Sodium carbonate.
Emanation apparatus.
Gamma scintillation counter.
Magnetic stirrer.
pH meter.
Plastic counting vials, 30-ml capacity.
Scintillation cell.
Vacuum pump.
Ethyl alcohol, absolute.
Hydrochloric acid.
Diethyl ether.
Drying oven.
Desiccator.
Low-background beta counter.
Vacuum filtration apparatus.
Strontium nitrate.
Ferric chloride.
Cerous nitrate.
Zirconyl chloride.
Sodium carbonate.
Sodium hydroxide.
Infrared lamp.
Ammonium acetate.
Ammonium acetate buffer, pH 5.

Ammonium oxalate.
Bromcresol green indicator.
Calcium nitrate.
Yttrium nitrate.
Hydrogen peroxide, 30%.
Fuming nitric acid.
Oxalic acid.
Lucite solution in acetone.
Glass-fiber filter paper.
Stainless steel planchets.

PROCEDURES

PROCEDURE 1: EXPERIMENTAL SAMPLE COLLECTION

1. Collect two 3-liter grab samples of tap water.
2. Label each container with proper information (see text).
3. Place a "radioactive" label on one sample.
4. To the sample with the "radioactive" label, add 0.01 μCi of ^{210}Pb, 0.01 μCi of ^{90}Sr, and 0.01 μCi of ^{140}Ba.
5. Record data in Data Table 20-5-1.

PROCEDURE 2: GROSS ALPHA AND GROSS BETA ACTIVITIES

1. Use both samples prepared in Procedure 1.
2. Examine samples according to procedure for gross alpha and gross beta activity, as given in the Subject Matter section of this module.
3. Record data and results in Data Table 20-5-2.

PROCEDURE 3: STRONTIUM IN SALINE WATER

1. Use both samples prepared in Procedure 1.
2. Examine samples according to procedure for Sr in saline water, as given in the Subject Matter.
3. Record data and results in Data Table 20-5-3.

PROCEDURE 4: DETERMINATION OF RADIOBARIUM

1. Use both samples prepared in Procedure 1.
2. Examine samples according to procedure for radiobarium, as given in the Subject Matter. NOTE: The procedure actually begins as a procedure for strontium, the oxalate precipitation method.
3. Record data in Data Table 20-5-4.

PROCEDURE 5: DETERMINATION OF RADIUM-226

1. Use a fresh sample of tap water for this procedure.
2. Examine sample according to the procedure for radium-226 given in the Subject Matter.
3. Record data and results in Data Table 20-5-5.

DATA TABLES

DATA TABLE 20-5-1
SAMPLE COLLECTION AND PREPARATION

Sample	Collected			Added		
	Volume	Date	Time	^{210}Pb	^{90}Sr	^{140}Ba
Nonradioactive						
Radioactive						

DATA TABLE 20-5-2
DETERMINATION OF GROSS ALPHA AND GROSS BETA ACTIVITY

Gross Alpha

Sample	Conductivity µMHO	Volume	Net cpm	pCi/l
Nonradioactive				
Radioactive				

Gross Beta

Nonradioactive				
Radioactive				

DATA TABLE 20-5-3
DETERMINATION OF STRONTIUM

Sample	Volume	Net cpm	pCi/1
Nonradioactive			
Radioactive			

DATA TABLE 20-5-4
DETERMINATION OF RADIOBARIUM

Sample	Volume	Barium Recovery	Date & Time Collected	Date & Time ^{140}La regrowth	Net	pCi/1
Nonradioactive						
Radioactive						

DATA TABLE 20-5-5
DETERMINATION OF RADIUM-226

Sample	Date & Time	Volume	Recovery	1st Removal Date & Time	2nd Removal Date & Time	Net cpm	pCi/1

REFERENCE MATERIALS

Health and Safety Laboratory *EML Procedures Manual, HASL-300, Revisions to August 1979*. New York: U.S. Energy Research and Development Administration, 1979.

APHA-AWWA-WPCF, *Standard Methods for the Examination of Water and Wastewater*, 13th ed. New York: American Public Health Association, Inc., 1971.

TEST

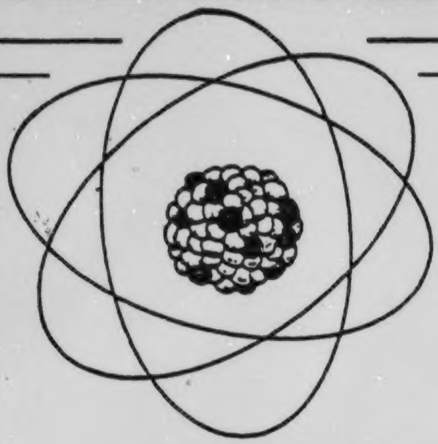
1. What should be the volume of a grab sample? What information should go on the label?

2. What should be the volume of the third portion of a 24-hour composite sample if flow is continuous at 90 liters/minute for the entire 24 hours and if the final volume desired in the composite is 2 liters?

3. Why is there a limitation on sample volumes to be evaporated for gross alpha and gross beta determinations? Why is the volume allowed for gross beta greater than that for gross alpha?

4. Why is the radon emanation method considered specific for radium-226?

5. Why is tritium in water "best" counted in a liquid scintillation counter? Would tritium be detected from the method in this module if the tritium were attached to an organic molecule? Why?



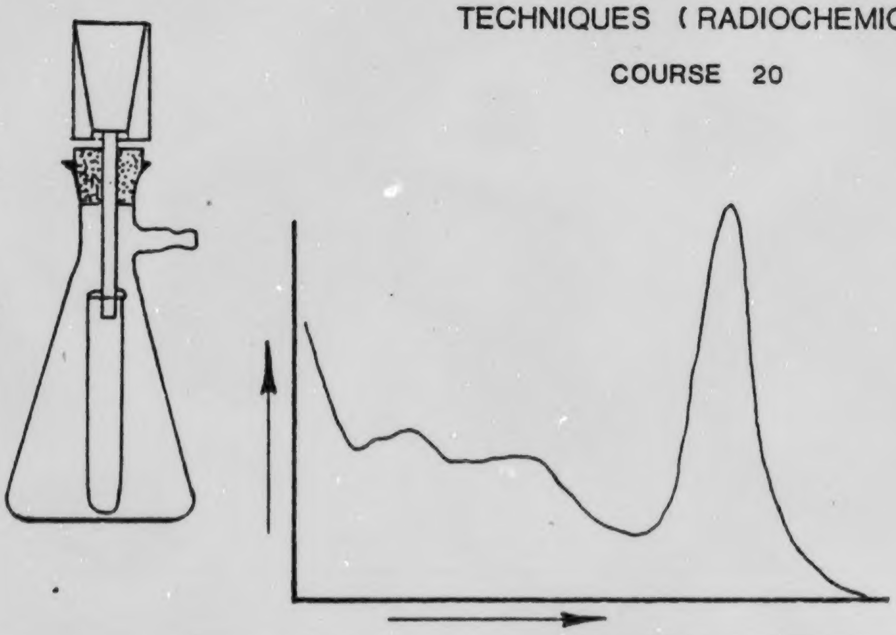
NUCLEAR TECHNOLOGY

MODULE 20-6

WATER MONITORING TECHNIQUES - SUSPENDED SOLIDS

RADIATION MONITORING
TECHNIQUES (RADIOCHEMICAL)

COURSE 20



CENTER FOR OCCUPATIONAL RESEARCH AND DEVELOPMENT

COURSE MODULES

COURSE 20 RADIATION MONITORING TECHNIQUES (RADIOCHEMICAL)

MODULE 20-1	Radiation Sample Preparation – Liquid
MODULE 20-2	Radiation Sample Preparation – Solid
MODULE 20-3	Air Monitoring Techniques – Particulate
MODULE 20-4	Air Monitoring Techniques – Gaseous
MODULE 20-5	Water Monitoring Techniques – Dissolved Solids
⇨ MODULE 20-6	Water Monitoring Techniques – Suspended Solids
MODULE 20-7	Radiation Monitoring of Biological Samples

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INTRODUCTION

The suspended solids in a water sample are those materials that are not dissolved. These materials usually are removed by filtration and may be referred to as "nonfilterable solids," which are solids that will not pass through the filter. The glass fiber filter is sufficiently fine for most determinations; however, since the amount of suspended matter removed by a filter varies with the porosity of the filter and the size of the particles being filtered, special-purpose filters occasionally may be required.

The radiation technician is interested in whether or not the suspended solids are themselves radioactive materials or if they have radioisotopes adsorbed onto them. This module provides the technician with filtration techniques, radiochemical preparation for filtration, and appropriate radioassay procedures for some routine procedures.

MODULE PREREQUISITES

The student should have completed a course in chemistry; Course 1, "Radiation Physics"; Course 11, "Radiation Detection and Measurement"; and Module 20-5, "Water Monitoring Techniques – Dissolved Solids."

OBJECTIVES

Upon completion of this module, the student should be able to:

1. Given a sample of water containing suspended matter, determine the concentration of suspended matter in the sample.
2. Prepare a sample of suspended solids for counting.
3. Determine the gross alpha activity per unit volume, utilizing the suspended solids in a water sample.
4. Determine the gross beta activity per unit volume, using the suspended solids in a water sample.
5. Analyze a sample of water for ^{226}Ra in the suspended solids.
6. Prepare a sample of suspended solids for gamma counting of a specific radionuclide.

The radiation technician easily can measure out a certain volume of water, evaporate it to dryness in a previously weighed dish, and then weigh the residue. However, the technician's life is not quite that simple. The technician must know whether or not the material is radioactive and, if so, which radioisotopes are present in what amount.

SUSPENDED SOLIDS

Before procedures are begun to determine the radioactivity present in suspended material, the concentration of the suspended material in the water sample must be known. Concentration is determined directly by the filtering of a measured sample of the water and the weighing of residue on the filter. The following procedure is a standard method.

1. Dry a fiberglass filter for 1 hour at 105°C.
2. Cool the filter in a desiccator.
3. Weigh on an analytical balance to 4 decimal places.
4. Place the fiberglass filter on a filter chimney (Figure 20-6-1).
5. Stir the water sample thoroughly.
6. Measure out 100 ml of the water sample into a graduated cylinder; pour into the filter chimney.
7. Filter the sample with suction.
8. Rinse the graduated cylinder at least 3 times with distilled water, pour the rinsings through the filter.
9. Remove the fiberglass filter, and dry it in a drying oven.
10. Cool the filter in a desiccator.
11. Weigh on an analytical balance to 4 places.

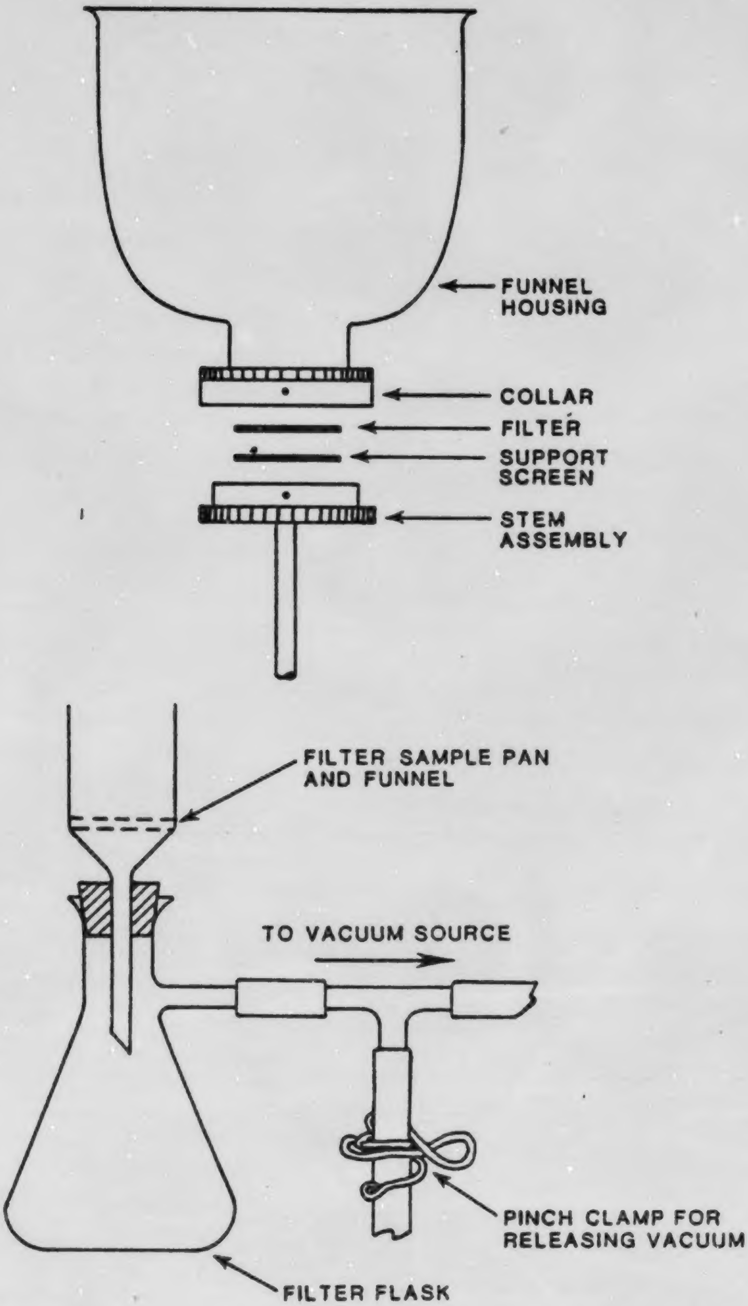

FUNNEL HOUSING
COLLAR
FILTER
SUPPORT SCREEN
STEM ASSEMBLY
FILTER SAMPLE PAN AND FUNNEL
TO VACUUM SOURCE
PINCH CLAMP FOR RELEASING VACUUM
FILTER FLASK

Figure 20-6-1. Chimney filters.

12. Repeat Steps 9 through 11 until the weights agree to within 0.2 mg.

13. Calculate the solids content from Equation 20-6-1.

$$\text{Suspended Solids (mg/ml)} = \frac{\text{Gain in Weight (mg)}}{\text{Volume Filtered (ml)}}$$

Equation 20-6-1
(Equation = Eq.)

EXAMPLE

Given: A 100-ml sample that produces an increase in weight of the filter of 24.5 mg.

Find: Amount of suspended solids, in mg/ml..

Solution: Use Equation 20-6-1:

$$\text{Suspended Solids (mg/ml)} = \frac{\text{Gain in Weight (mg)}}{\text{Volume Filtered (ml)}}$$

$$= \frac{24.3\text{ mg}}{100\text{ ml}}$$

$$\text{Suspended Solids (mg/ml)} = 0.243\text{ mg/ml.}$$

The amount of suspended solids present must be determined before a sample of the appropriate size can be chosen for radiation determination. If a sample of the wrong size were chosen, problems would arise because of self-absorption.

GROSS ALPHA AND BETA ACTIVITIES IN SUSPENDED SOLIDS

A water sample may contain microscopic living organisms or other forms of suspended material with which substantial amounts of radioactive materials may be associated. This is particularly true of industrial effluent, which may have a large portion of its total radioactivity in the suspended solids. Since the ultimate effect of suspended radioactivity on the environment is different from that of dissolved material, it is important to measure this form of radioactive contamination.

The method of separation is similar to the previous procedure for suspended solids, except that the sample must be proportioned in order to provide a final filter increase in weight of not more than 5 mg/cm^2 for alpha counting and not more than 10 mg/cm^2 for beta counting. Filtration is best accomplished on a Millipore type HA membrane filter, or equivalent, as follows:

Procedure

1. Measure the inside diameter of the chimney filter; calculate the surface area of the filter that will be covered.
2. Use previously determined solids concentration and area of the filter to be used to calculate the volume of sample required to give a dry solids deposit of 5 mg/cm^2 for gross alpha or 10 mg/cm^2 for gross beta activity, according to Equations 20-6-2 and 20-6-3.

$$\text{TSL (mg)} = \text{DTL (mg/cm}^2\text{)} \times \text{AF (cm}^2\text{)} \qquad \text{Eq. 20-6-2}$$

where:

TSL = total solids limit in mg.
DTL = density thickness limit of 5 mg/cm^2 for alpha counting and 10 mg/cm^2 for beta counting.
AF = area of the filter that will be used.

$$\text{VS (ml)} = \frac{\text{TSL (mg)}}{\text{SC (mg/ml)}} \qquad \text{Eq. 20-6-3}$$

where:

VS = volume of sample to be filtered.
TSL = total solids limit in mg.
SC = suspended solids concentration in mg/ml.

EXAMPLE

Given: Useful area of filter is 10 cm^2, and water sample contains 0.243 mg/ml of suspended solids.

Find: Volume of a water sample that must be filtered in order to prepare a sample for gross beta counting.

Solution: From Equation 20-6-2 –

$$TSL = DTL \text{ x } AF$$

$$TSL \text{ (mg)} = 10 \text{ mg/cm}^2 \text{ x } 10 \text{ cm}^2$$

$$TSL = 100 \text{ mg.}$$

From Equation 20-6-3 –

$$VS \text{ (ml)} = \frac{TSL}{SC}$$

$$VS \text{ (ml)} = \frac{100 \text{ ml}}{0.243 \text{ mg/ml}}$$

$$VS = 411 \text{ ml.}$$

3. Dry and weigh a membrane filter to the nearest 0.1 mg.
4. Place the weighed membrane filter on the chimney filter.
5. Measure required volume of sample as calculated in Step 2.
6. Pour the measured sample through the filter, using gentle suction.
7. Rinse measuring container at least 3 times, using 15 to 20 ml of distilled water each time and pouring rinsings through the filter.
8. Remove filter and place it on a tared planchet and allow to air dry.
9. Spread 1 ml of ether-acetone reagent over the filter to cause the membrane to adhere to the planchet, then air-dry.
10. Add just enough conducting fluid to the solids on the filter to moisten them slightly.
11. Dry in a drying oven at 105°C for one hour, cool in a desiccator; and weigh to the nearest 0.1 mg.

12. Repeat Step 11 until successive weighings indicate a constant weight.
13. Count gross alpha and gross beta activities in an internal proportional counter.
14. Calculations: Gross alpha and gross beta activities usually are reported as picocuries (pCi) per liter of water.

$$\text{Gross alpha activity (pCi/l)} = \frac{C_a}{E_a \times V \times F_{sa}} \qquad \text{Eq. 20-6-4}$$

where:

C_a = net alpha count rate (cpm).
E_a = efficiency for counting alpha activity (cpm/pCi).
V = volume of the water sample in liters (l).
F_{sa} = correction factor for self absorption.

$$\text{Gross beta activity (pCi/l)} = \frac{C_b}{E_b \times V \times F_{sa}} \qquad \text{Eq. 20-6-5}$$

where:

C_b = net beta count rate (cpm).
E_b = efficiency for counting beta activity (cpm/pCi).
V = volume of water sample in liters (l).
F_{sa} = correction factor for self absorption.

EXAMPLE

Given: A sample of water, 411 ml of which gives a count rate of 42 cpm on an instrument with a beta efficiency of 0.98 cpm/pCi and a self absorption factor of 0.77.

Find: Gross beta activity in pCi/l.

Solution: Use Equation 20-6-5:

$$\text{Gross beta activity} = \frac{C_b}{E_b \times V \times F_{sa}}$$

$$= \frac{42 \text{ cpm}}{0.98 \text{ cpm/pCi} \times 0.411 \times 0.77}$$

$$\text{Gross beta activity} = 135.4 \text{ pCi/l.}$$

Occasionally a sample prepared for alpha counting may exceed 10 mg/cm^2. When this occurs, the sample may be assumed to be infinitely thick, and the results may be reported as an "approximate" gross alpha assay. However, if precise results are needed, the test must be repeated.

When a gross beta activity sample exceeds 20 mg/cm^2 the strontium-90/yttrium-90 self-absorption curves may be employed. The results then must be reported as beta-gamma activity and not gross beta activity. It is possible to use the sample prepared for alpha counting to determine beta activity if the appropriate self-absorption curves are available.

RADIUM-226 IN SUSPENDED SOLIDS

Radium-226 in suspended solids is determined indirectly by the radon-222 emanation method. (A de-emanator is illustrated in Figure 20-6-2.) A water sample of 50 to 1000 ml is filtered through a fine cellulose filter, such as a Whatman No. 42; is dried, ashed, and then fused with sodium carbonate in a platinum crucible. The fused material is dissolved in hydrochloric acid; barium carrier is added; and silica is removed by filtration.

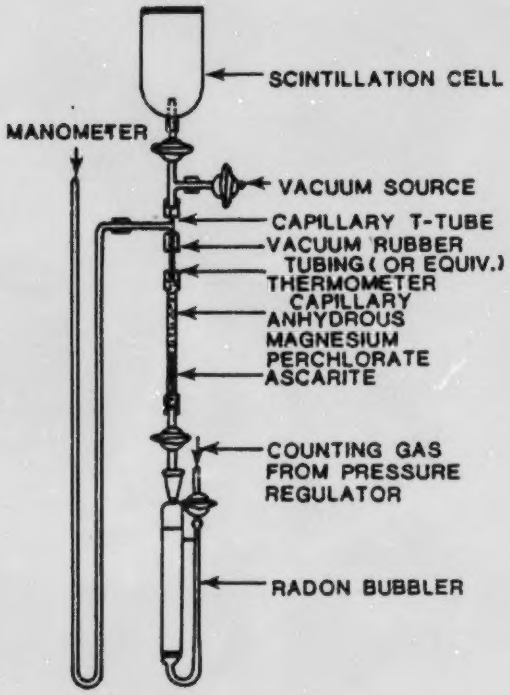

SCINTILLATION CELL
MANOMETER
VACUUM SOURCE
CAPILLARY T-TUBE
VACUUM RUBBER TUBING (OR EQUIV.)
THERMOMETER CAPILLARY
ANHYDROUS MAGNESIUM PERCHLORATE
ASCARITE
COUNTING GAS FROM PRESSURE REGULATOR
RADON BUBBLER

Figure 20-6-2. Radon gas de-emanator.

The filtrate is transferred to a radon bubbler, and radon is withdrawn to give an analysis for radium, as was done for dissolved solids. The sodium carbonate fusion is necessary to remove the silica that usually is present in suspended material. The barium carrier is added to ensure the complete precipitation of radium.

There is a variety of procedures for determination of radium-226 by radon-222 emanation. Some of these procedures require a minimum of chemistry but have inherent physical problems; others depend upon chemical separation, concentration, and purification of the radium before de-emanation and counting. The following method requires only a moderate amount of chemistry to increase sensitivity, and has the added advantage of using highly sensitive alpha scintillation counting.

The chemical separation and concentration technique employed depends upon the similarity of the chemistry of barium and radium. Chemical similarity permits the use of a considerable amount of barium as a carrier for radium coprecipitation. It must be remembered, however, that all barium salts contain some radium-226, and that, therefore, reagent testing must be done to correct for the radium introduced by the reagents.

The only radionuclides that could interfere with this procedure rarely are found in water unless it is in contaminated with uranium or thorium in greater than trace amounts. Additionally, they have very short half-lives, on the order of seconds; hence, only their alpha-emitting daughters would interfere. There is very little interference from stable elements.

The minimum detectable limit of this procedure should be on the order of 0.05 pCi of radium-226.

Procedure

1. Dry and weigh a Whatman No. 42 filter paper to the nearest 0.1 mg.
2. Place the filter paper on a chimney filter.
3. Measure a volume of sample to be tested in such a manner that the filtered residue will not exceed 1000 mg. Usually, a residue of 100 to 300 mg is sufficient; however, more or less may be used. In any case, the volume of water filtered should not exceed 1 liter.
4. Filter the sample through the filter paper, using gentle suction.
5. Rinse the measuring container at least 3 times with 15-ml portions of distilled water. NOTE: The filtrate can be saved for the determination of dissolved radium, if desired.
6. Remove the filter from the chimney funnel and dry in a drying oven for 1 hour at 105°C.
7. Cool the dried filter in a desiccator, and weigh the filter to determine the weight of suspended solids.
8. Place the filter paper with the residue in a platinum crucible and ignite in a muffle furnace at 500°C for 1 hour.
9. Remove the crucible and residue from the furnace and allow to air-cool.
10. Add anhydrous sodium carbonate to the residue in the crucible in the ratio of 3 g of sodium carbonate to 1 g of residue. NOTE: The weight of residue to be used here was determined in Step 7.
11. Use a glass stirring rod to mix the residue and the sodium carbonate thoroughly.

12. Fuse the mixture at 900°C in a muffle furnace for 1 hour.
13. Remove the crucible from the muffle furnace, and allow the melt to air-cool.
14. Add 30 ml of 4N hydrochloric acid to the residue in the crucible and place on a hot plate until the liquid just begins to boil.
15. Quantitatively transfer the solution from the crucible to a clean 400-ml beaker, rinsing the crucible at least 3 times with distilled water, and adding the rinsings to the solution in the beaker.
16. Dilute the solution in the beaker to approximately 250 ml with distilled water.
17. Add 2 ml of 50% ammonium sulfate, 20 ml of 10% disodium EDTA, 38 mg of barium nitrate, and 0.02 μCi of barium-133.
18. Adjust the pH to 4.6, and stir with a magnetic stirrer for 5 minutes.
19. Allow to stand for 1 hour or, preferably, overnight.
20. Decant as much of the supernate as possible, discarding the decantate.
21. Quantitatively transfer the precipitate to a 50-ml centrifuge tube, using distilled water and filling the tube to the 50-ml mark.
22. Mix well with a glass stirring rod. Centrifuge for 10 minutes; and decant, discarding the decantate.
23. Add 30 ml of 3N sodium carbonate, and heat in a boiling-water bath for 30 minutes, stirring with a glass stirring rod every 5 minutes.
24. Remove from the water bath and allow to air-cool. Centrifuge and decant, discarding the decantate.

25. Add 2 ml of 16N nitric acid to dissolve the precipitate. NOTE: If the precipitate does not completely dissolve, transfer the solution to a plastic container, and repeat Steps 23 through 25 with the undissolved residue.
26. When the material is completely dissolved, combine the solutions into the plastic container and dilute to 25-ml total volume with distilled water.
27. Count the activity of the barium-133 in a gamma scintillation counter, and calculate the barium recovery by dividing the measured activity of the solution by the measured activity of 25 ml of water containing 0.02 μCi of barium-133.
28. Transfer the solution to a radon bubbler, then de-emanate the solution by slowly drawing air through the bubbler for 2 minutes.
29. Evacuate the bubbler for 3 minutes, then seal and store for 2 to 4 weeks to allow for radon-222 growth.
30. After 2 to 4 weeks, prepare an absorption tube with magnesium perchlorate in the upper half and ascarite in the lower half.
31. Attach the absorption tube to the bubbler and the vacuum pump, and the scintillation cell to the absorption tube.
32. Evacuate the absorption tube and the scintillation cell for at least 5 minutes; then seal the system.
33. Open all the stopcocks between the scintillation cell and the bubbler, keeping the system sealed.
34. Slowly open the lower stopcock of the radon bubbler, allowing air to bleed into the system until the pressure is at equilibrium.
35. Close the stopcock to the scintillation cell, and record the time as the beginning of radon-222 decay.
36. Remove the scintillation cell, and reseal the bubbler system.

37. Store the scintillation cell for 2 hours; measure the alpha activity of the cell in a radon counter. (Figure 20-6-3.) Record the time of counting to determine the correction factor for the growth of radon-222 daughters.

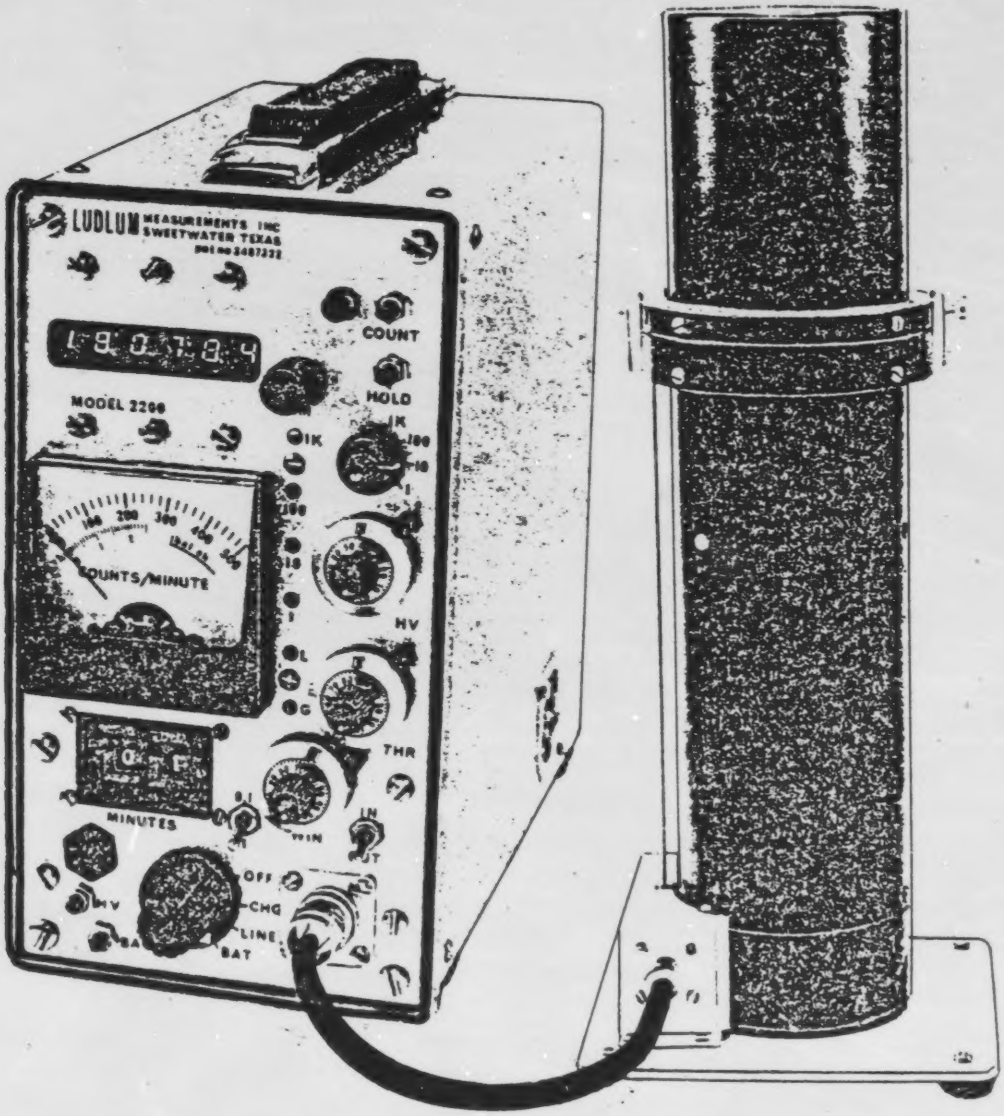

LUDLUM MEASUREMENTS INC
SWEETWATER TEXAS
COUNT
HOLD
MODEL 2200
COUNTS/MINUTE
HV
THR
MINUTES
WIN
OFF
CHG
LINE
BAT

Figure 20-6-3. A radon flask counting system.

38. Calculate radium-226 activity according to Equation 20-6-6.

$$\underset{\text{(pCi/l)}}{{}^{226}\text{Ra activity}} = \frac{C_a}{V \times E \times F_1 \times F_2 \times R} \quad \text{Eq. 20-6-6}$$

where:

C_a = net alpha count rate in cpm.

V = volume of sample in liters.

E = efficiency of de-emanation and counting in cpm/pCi.

F_1 = correction factor $1-e^{-\lambda t}$ for radon-222 growth, where t is the time from the first de-emanation of radon to the end of the second de-emanation.

F_2 = correction factor of $1-e^{-\lambda t}$ for radon daughter ingrowth, where t is the time from the end of the second de-emanation of radon to the time of counting.

R = recovery fraction of the barium carrier.

ORGANIC SOLIDS

Occasionally, samples that have the radioactivity bound to organic materials or living organisms in the sample will be encountered. When this occurs, some method must be used to convert the sample into a soluble form. The procedure usually employed is an acid digestion called "wet-ashing."

Procedure

1. Desiccate and weigh to the nearest 0.1 mg a membrane filter. Millipore Type HA or equivalent.
2. Place the filter in a chimney funnel.
3. Filter, with gentle suction, sufficient water sample to provide a deposit of approximately 5 mg/cm^2.
4. Remove the membrane filter with its cake of suspended matter; desiccate and weigh to the nearest 0.1 mg. Redesiccate and reweigh.
5. Place filter and sample in a clean 400-ml beaker and cautiously add 20 ml of concentrated nitric acid.
6. Place the beaker on a hot plate; and gently heat to boiling. NOTE: This should be done under a fume hood.
7. Evaporate the solution to a volume of approximately 10 ml.
8. Carefully add 15 ml of fuming nitric acid. Cover the beaker with a watch glass and boil for 20 minutes.
9. Remove the watch glass, and evaporate to a volume of approximately 10 ml.
10. Repeat Steps 8 and 9.
11. Add 100 ml of concentrated nitric acid, and evaporate to a volume of 25 to 30 ml.
12. Quantitatively transfer to a 50-ml centrifuge tube; then centrifuge for 5 minutes.
13. Decant into a clean, dry 150-ml beaker and save.
14. Quantitatively wash the precipitate into a platinum dish, using distilled water.
15. Add 20 ml of concentrated hydrofluoric acid, and slowly evaporate to dryness.
16. Allow to air-cool; then add 30 ml of concentrated hydrofluoric acid, and slowly evaporate to dryness.

17. Repeat Step 16.
18. Allow to air-cool; add 10 ml of distilled water and 10 ml of concentrated nitric acid; and boil for 10 minutes, adding a few ml of water if needed to prevent dryness.
19. Quantitatively transfer to the beaker containing the decantate from Step 13.
20. The sample now may be diluted to a known volume and subjected to radiochemical analysis, or it may be treated as a sample of dissolved solids.

This procedure will produce a solution from most samples without the use of perchloric acid. If the procedure does not produce a solution, a perchloric acid procedure may be used after Step 19. DANGER: The use of perchloric acid should be attempted only as a last resort, under an approved perchloric acid fume hood, and under close supervision by a person familiar with perchloric acid. There is an extreme danger of explosion when the acid contacts organic material.

GAMMA COUNTING OF SUSPENDED MATTER

The preparation of suspended material for gamma analysis appears quite simple on the surface; however, there are several important points to remember, the omission of any one of which may render the sample useless.

The first of these points for consideration is sample size. Since correction for sample self-absorption of gamma rays is unnecessary, one could get the impression that the thickness of the solid material to be deposited on a filter is of no consequence. However, absorption of beta and alpha particles by the material will add bremsstrahlung to the gamma count.

A second part is in the use of organic solvents to speed drying. Part of the radioactive material of interest may be fatty tissue or oils that could be soluble in alcohol, ether, or acetone commonly utilized in the drying of filtered samples. Therefore, these materials should not be employed.

Although the filter media of choice is a membrane filter, the pores of this material may become rapidly clogged. The second choice is fibrous-glass filter paper. This material usually provides excellent results and is considered by some to be the material of first choice.

The samples probably are best mounted in plastic planchets, since the plastic will absorb both alpha and beta radiations and produce the least amount of bremsstrahlung.

Procedure

1. Desiccate and weigh a filter to the nearest 0.1 mg; mount filter on a chimney filter.
2. Use the suspended solids concentration previously determined to determine the quantity of sample necessary to produce a deposit on the filter of approximately 20 mg/cm^2.
3. Measure the quantity of well-stirred water sample necessary; filter, with gentle suction, through the filter.
4. Rinse the deposit three times with 10-ml portions of distilled water.
5. Remove the filter, and desiccate to a constant weight, weighing to the nearest 0.1 mg.

6. Mount the sample in a plastic planchet.
7. Gamma count or analyze with a gamma spectrometer.

RADIOCHEMICAL CESIUM-137

The presence of fission products may be determined by the identification and confirmation of ^{137}Cs. The presence may be suspected by appearance of the cesium gamma peak at 0.662 MeV when suspended material is analyzed as in the previous procedure. Presence of fission products can be confirmed by the following procedure.

Cesium-137 and a cesium carrier are equilibrated by digestion of the carrier with a sample suspected of containing cesium in nitric acid. The alkaline earths, rare earths, and iron are removed by scavenging with calcium carbonate. Ammonium phosphomolybdate then is used to extract cesium, rubidium, and potassium from the solution. The cesium can be determined by gamma counting of the precipitate or can be further purified by ion exchange to determine an even lower activity.

Since cesium is a volatile material at high temperature, the suspended solids should be separated and treated by the wet-ashing method – the procedure for organic solids. If the sample is so large as to prohibit wet ashing, it can be ignited in a muffle furnace; however, the ignition temperature must not exceed 900°F. Regardless of the procedure used, the sample should be in a nitric acid solution ready to proceed.

Procedure

1. Adjust solution to a pH of 5 to 6 with sodium hydroxide.
2. Add 200 ml of a saturated solution with sodium carbonate, and stir for 15 minutes.
3. Allow the precipitate to settle for 5 minutes.
4. Filter, with suction, through a Whatman No. 40 filter paper, washing the precipitate three times with 10-ml portions of 10% sodium carbonate and saving the filtrate.
5. Add hydrochloric acid to the filtrate to a pH of 1; then add 100 ml of concentrated hydrochloric acid, and stir for 15 minutes.
6. Allow to stand for 15 minutes. If any precipitate forms, filter through a glass fiber filter, washing the precipitate twice with 10-ml portions of 6N hydrochloric acid.
7. To the filtrate from No. 6 or the solution from No. 5, add 1 gram of ammonium phosphomolybdate; and stir for 15 minutes.
8. Allow the precipitate to settle; decant off as much of the supernate as possible.
9. Quantitatively transfer to a 50-ml centrifuge tube, and centrifuge for 5 minutes.
10. Wash the precipitate 3 times with distilled water made acidic by the addition of 5 drops of hydrochloric acid.
11. If the precipitate is to be counted, quantitatively transfer to a glass fiber filter; dry, mount, and count. NOTE: Beta counting may be done immediately. The sample should be stored for 2 hours before gamma counting.
12. If the precipitate is to be distilled further, add 10 ml of distilled water; then add 8N sodium hydroxide dropwise, with constant stirring, until the precipitate is in solution.

13. Add 10 ml of tetrasodium EDTA and stir.
14. Pass the sample through a prepared column of Bio-Rex 40 that contains at least 15 ml of the resin at a flow rate not exceeding 1 ml per minute, discarding the effluent.
15. Pass 100 ml of water through the column at a flow rate not exceeding 2 ml per minute, discarding the effluent.
16. Prepare a solution of 10 ml of concentrated hydrochloric acid in 990 ml of distilled water. Pass this solution through the column at a rate of 5 ml per minute, discarding the effluent.
17. Pass 200 ml of 1N hydrochloric acid through the column at a flow rate not exceeding 5 ml per minute, collecting the eluate in a 400-ml beaker.
18. Evaporate the eluate in the beaker to dryness.
19. Quantitatively transfer the sample from the beaker to a 50-ml centrifuge tube, using distilled water made acidic by the addition of 5 drops of hydrochloric acid.
20. Add 10 ml of 10% chloroplatinic acid solution with continuous stirring; continue to stir for 10 minutes.
21. Refrigerate at 5°C overnight; then filter with suction through a Whatman No. 42 filter paper.
22. Wash the precipitate on the filter paper once with water and twice with ethanol.
23. Dry the filter, and precipitate for 1 hour at 105°C.
24. Cool in a desiccator for 1 hour.
25. Beta count or gamma count on a pulse height analyzer adjusted to count 0.662-MeV gamma rays.
26. Calculations must be based upon whether the sample is beta counted or gamma counted. Either method is acceptable, but gamma counting may be the better procedure.

EXERCISES

1. Outline a procedure by which the total suspended solids in a sample of water can be determined.
2. Write a procedure for the preparation of a sample of water that contains suspended matter for gamma counting.
3. Why would a determination of the amount of ^{137}Cs in suspended matter be useful enviromental information?
4. Why are samples of suspended solids prepared for alpha counting limited to a thickness not to exceed 5 mg/cm^2?
5. Why is the emanation method the preferred procedure for the determination of radium-226?
6. List radioisotopes and their parents and daughters that could interfere with the emanation procedure for determination of radium-226.
7. What should be done if a sample of suspended solids prepared for gross beta counting is found to have a density thickness of 14 mg/cm^2?
8. Write the formula used to calculate the concentration of suspended matter in water.
9. Why is it more useful to calculate suspended solids concentration in mg/ml rather than the more usual mg/l?
10. Outline a procedure for the wet ashing of a sample that contains suspended organic materials.

MATERIALS

Glass fiber filter paper.
Desiccator.
Silica gel.
Analytical balance.
Magnetic stirrer.
Mechanical stirrer.
100-ml graduated cylinder.
Chimney filter.
Vacuum pump.
Vacuum flask.
Drying oven.
Calipers.
Planchets, stainless steel, plastic.
Acetone.
Ether.
Conducting fluid for proportional counter.
Whatman No. 42 filter paper.
Timer.
Platinum crucible.
Platinum dish.
Sodium carbonate, anhydrous.
Hydrochloric acid, concentrate, 4N.
50% ammonium sulfate solution.
10% disodium EDTA solution.
Barium nitrate.
0.1 µCi barium-133.
pH meter.
Centrifuge.
50-ml centrifuge tubes.
3N sodium carbonate solution.
16N nitric acid.
Plastic containers, 250 ml.

Radon emanator.
Magnesium perchlorate.
Ascarite.
Radon scintillation cell.
Membrane filters, Millipore type HA.
Beakers, 150, 250, 400, 500, 1000 ml.
Volumetric pipettes.
Hot plate.
Fume hood.
Fuming nitric acid.
Concentrated hydrofluoric acid.
Sodium hydroxide.
Sodium carbonate, saturated solution.
Whatman No. 40 filter paper.
Ammonium phosphomolybdate.
8N sodium hydroxide.
Tetrasodium EDTA.
Bio-Rex 40, 100 mesh ion-exchange resin.
Proportional counter.
Radon scintillation counter.
Pulse height analyzer, scintillation counter.
100-channel pulse height analyzer equipped with a 3 x 3 NaI (TL) crystal.

PROCEDURES

Obtain a sample of water that contains suspended solids, with a total volume of 4 ml. The sample should be spiked with 0.01 μCi of ^{226}Ra and 2 μCi of ^{137}Cs.

PROCEDURE 1: DETERMINATION OF SUSPENDED SOLIDS

1. Follow the procedure given in the Subject Matter for determination of suspended solids.
2. Record results in Data Table 20-6-1.

PROCEDURE 2: GROSS ALPHA ACTIVITY IN SUSPENDED SOLIDS

1. Follow the procedure given in the Subject Matter for determination of gross alpha activity in suspended solids.
2. Report results in Data Table 20-6-2.

PROCEDURE 3: GROSS BETA ACTIVITY IN SUSPENDED SOLIDS

1. Follow the procedure given in the Subject Matter for determination of gross beta activity in suspended solids.
2. Report results in Data Table 20-6-3.

PROCEDURE 4: DETERMINATION OF RADIUM-226 IN SUSPENDED SOLIDS

1. Follow the procedure given in the Subject Matter for determination of radium-226 in suspended solids.
2. Report results in Data Table 20-6-4.

PROCEDURE 5: PREPARATION OF SAMPLES CONTAINING ORGANIC SOLIDS

1. Prepare a sample from the water sample by measuring 500 ml of water and adding 2 ml of milk to the water.
2. Follow procedure given in the Subject Matter for a sample containing organic solids.
3. After the sample is in solution, prepare samples for gross alpha and gross beta analysis according to the procedures given in Module 20-5, "Water Monitoring Techniques – Dissolved Solids."
4. Determine the gross alpha and beta activities of the samples.
5. Record data in Data Table 20-6-5, and compare with Data Tables 20-6-2 and 20-6-3.

PROCEDURE 6: GAMMA COUNTING OF SUSPENDED MATTER

1. Prepare a sample for gamma counting according to the procedure given in the Subject Matter for the gamma counting of suspended matter.
2. Count the sample on a 100-channel pulse height analyzer, set for 1.5 MeV/100 ch.
3. Plot the spectrum in Figure 20-6-4.

PROCEDURE 7: CESIUM-137 IN SUSPENDED MATTER

1. Prepare a sample according to the procedure for cesium in suspended matter.
2. Count both beta and gamma activities.
3. Record data in Data Table 20-6-6.

DATA TABLES

DATA TABLE 20-6-1

SUSPENDED SOLIDS

Sample Volume (ml)	Wt of Filter and Sample (mg)	Wt of Filter (mg)	Wt of Sample (mg)	Concentration of Solids (mg/ml)

DATA TABLE 20-6-2

GROSS ALPHA ACTIVITY IN SUSPENDED SOLIDS

Volume of Sample (ml)	Area of Filter (cm^2)	Wt of Sample (mg)	Density Thickness (mg/cm^2)	Activity of Sample (pCi/l)

DATA TABLE 20-6-3

GROSS BETA ACTIVITY IN SUSPENDED SOLIDS

Volume of Sample (ml)	Area of Filter (cm^2)	Wt of Sample (mg)	Density Thickness (mg/cm^2)	Activity of Sample (pCi/l)

DATA TABLE 20-6-4
RADIUM-226 IN SUSPENDED SOLIDS

Volume of Sample (ml)	Wt of Solids (mg)	Ba-133 Sample (cpm)	Activity Standard (cpm)	%Ba Recovery	Emanation Date/Time 1st 2nd	Count Date Time	Activity (pCi/l)

DATA TABLE 20-6-5
SAMPLES CONTAINING ORGANIC SOLIDS

Radiation Type	Volume of Sample (ml)	Area of Filter (cm^2)	Wt of Sample (mg)	Density Thickness (mg/cm^2)	Activity of Sample (pCi/l)

DATA TABLE 20-6-6
CESIUM-137 IN SUSPENDED MATTER

Volume Sample (ml)	Wt of Solids (mg)	Activity of ^{137}Cs (pCi/l)

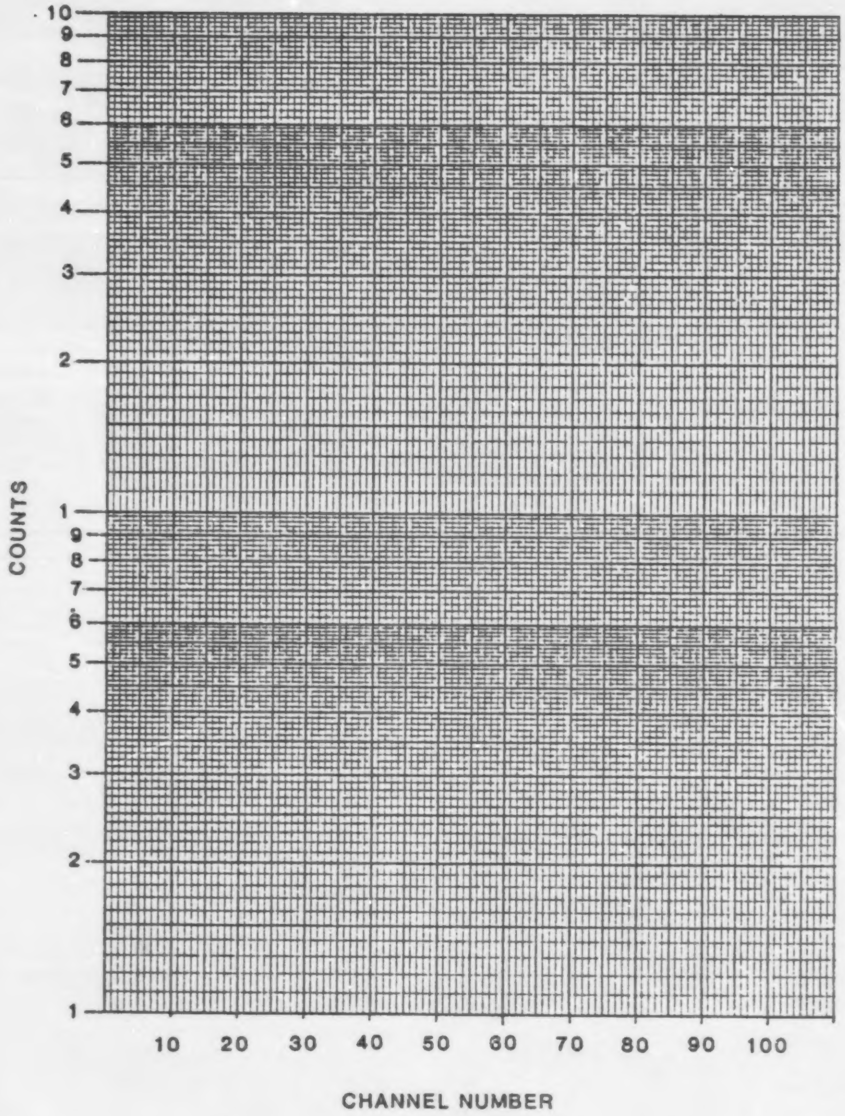


Figure 20-6-4. Gamma counting of suspended matter sample spectrum.

REFERENCE MATERIALS

APHA-AWWA-WPCF, Standard Methods for the Examination of Water and Wastewater, 13th edition, New York: American Public Health Association, Inc. 1971.

HEALTH AND SAFETY LABORATORY, EML Procedures Manual, HASL-300, Revisions to August 1979, New York: U.S. Energy Research and Development Administration, 1979.

TEST

1. A 100-ml sample of water was found to have a suspended solids content of 97 mg. What is the suspended solids concentration in mg/ml?

2. Outline a procedure for determining the gross alpha and gross beta activity of the suspended solids in a water sample.

3. List the isotopes that could interfere with the emanation method of determining radium-226, and the reasons why they do not interfere.

4. Outline a procedure for separating cesium-137 from suspended solids.

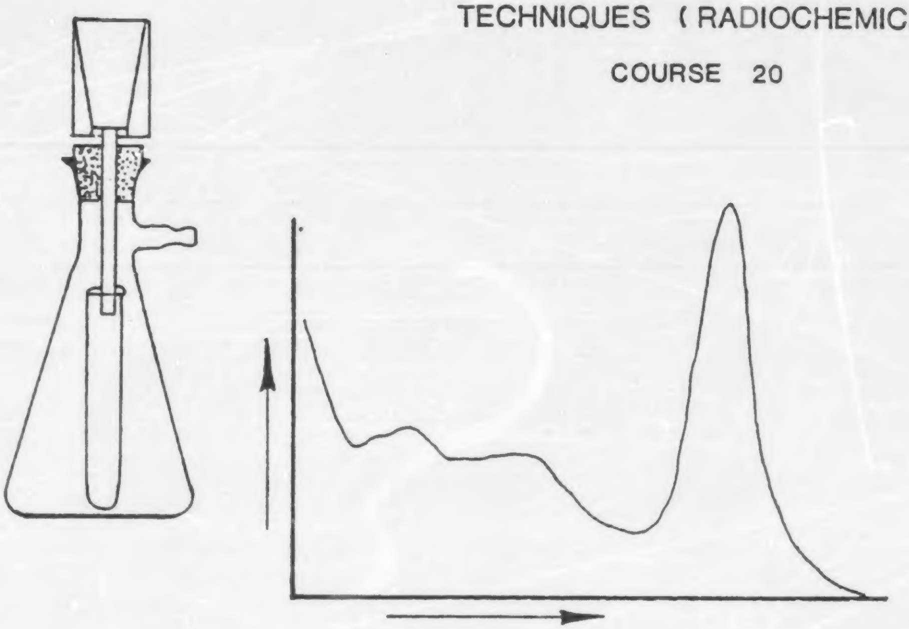
5. Outline a procedure for preparing a sample of suspended solids for gamma counting.

NUCLEAR TECHNOLOGY

MODULE 20 - 7

RADIATION MONITORING OF BIOLOGICAL SAMPLES

RADIATION MONITORING
TECHNIQUES (RADIOCHEMICAL)
COURSE 20



CORD CENTER FOR OCCUPATIONAL RESEARCH AND DEVELOPMENT

COURSE MODULES

COURSE 20 RADIATION MONITORING TECHNIQUES (RADIOCHEMICAL)

MODULE 20-1	Radiation Sample Preparation — Liquid
MODULE 20-2	Radiation Sample Preparation — Solid
MODULE 20-3	Air Monitoring Techniques — Particulate
MODULE 20-4	Air Monitoring Techniques — Gaseous
MODULE 20-5	Water Monitoring Techniques — Dissolved Solids
MODULE 20-6	Water Monitoring Techniques — Suspended Solids
MODULE 20-7	Radiation Monitoring of Biological Samples

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INTRODUCTION

Environmental samples often are collected around a nuclear facility. The samples are analyzed and a record kept of their characteristics. Some actual sample material should be retained for future analysis.

This module is an introduction to some of the techniques employed by technicians who work with biological materials. The scope is limited to techniques that the nuclear technician should find useful for routine analysis of environmental samples and other biological materials to determine radiological contamination. Most of the procedures may be adapted to materials other than those specified; however, the technician is cautioned to seek additional information if there is the least doubt concerning the adaptation of a procedure.

MODULE PREREQUISITES

The student should have completed Course 1, "Radiation Physics"; Course 11, "Radiation Detection and Measurement"; and the preceding modules of this Course.

OBJECTIVES

Upon completion of this module, the student should be able to:

1. Collect a sample of plant matter for radioassay.

2. Given a sample of plant material, determine the specific activity of the radioactive material present.
3. Given a sample of milk containing ^{90}Sr, determine the ^{90}Sr content in μCi/l.

SUBJECT MATTER

Methods suitable for the specific analysis of a radionuclide are dependent upon the characteristics of the element involved, the nature of the sample, and the degree of accuracy and precision required. This module is concerned with some of the basic techniques fundamental to the radioassay of biological samples.

Investigation of biological material requires the almost constant attention of the technician, especially during the ashing procedures. Because of this requirement, mass production operations are not recommended. Conversion of the biological material to a soluble material is critical to most procedures. Dry ashing is the least demanding of the technician's time and attention, but samples that contain radionuclides that volatilize at dry-ashing temperatures cannot be treated in this manner. Wet ashing, or acid digestion, while useful for volatile material, requires almost constant attention by the technician for at least several hours. Usually, no more than four samples can be wet ashed by a technician in a single day.

Samples of materials that have a high silica content are best treated by fusion. Again, volatilization temperatures may present problems. Some samples can be solubilized by acid digestion in conjunction with treatment with hydrofluoric acid to dissolve the silica. The technician always must bear in mind that the radionuclide to be analyzed determines to some extent, the techniques to be used in preparation of the sample.

SAMPLE COLLECTION

Since there is such a variety of biological material, there is a variety of sampling techniques and procedures. These techniques and procedures are controlled by variables, not the least of which are the purpose of sampling, the elements sought, or the material being sampled. There are, however, a few general criteria that are common.

The proper collection of a sample is just as important as the reliability of any analytical procedure that is to follow. An improperly collected sample is almost useless. The sample should be truly typical of the material that it represents. This, in turn, is affected by the purpose of sampling. For instance, plant material may be sampled for a variety of reasons. The technician who samples plant material must remember that new growth, near the top of the plant, represents the most recent conditions. Additionally, samples collected near the surface of the ground may be contaminated with soil.

All work with biological samples must be done with extreme care to prevent cross contamination. Care is necessary because biological samples not only have all of the problems associated with inorganic samples, but also exhibit problems of cross contamination unique to each system.

It is not unusual to find very refined or sophisticated techniques of analysis applied to samples of dubious quality. It is unfortunate that, while it is possible to establish very accurate and precise procedures for radiometric and chemical analysis, such procedures for sampling do not exist. Statistical methods have been developed for some materials, but procedures must be adapted for each given set of circumstances.

Samples of materials for radiochemical analysis may be analyzed as individual samples, or they may be combined to produce composite samples to represent a group of materials, locations, times, or a combination of these.

Most radiochemical analyses are performed to evaluate a possible hazard. A permissible level or a working level usually is established for routine surveys of a given radionuclide in a particular type of sample. Concentrations below a certain fraction of a permissible level (usually 10% or 25%) commonly are considered to be of no interest. The establishment of these levels helps to establish sampling procedures, details, and the sensitivity of analytical methods required.

A factor often overlooked is the necessity of retaining samples of materials required to document existing conditions. All too frequently, environmental samples are collected around a nuclear installation, analyzed, and the data recorded as documentation of conditions. However, if a portion of the samples is not stored for future use, there will be no method of obtaining needed information should the need arise to consider material that was not sought in the original analysis. At least a portion of each sample should be stored for possible future use. Storage is particularly important for samples collected for preoperational surveys.

The use of statistical methods is necessary in the establishment of sampling plans for both environmental and bioassay surveys. Initial sampling may be commenced from a set of random tables; but as soon as variability is established, application of statistical techniques probably will greatly reduce the number of samples required. Reduction in samples, in turn, will reduce the laboratory load and strain on the technician.

SAMPLE HANDLING

Often, a sample collected and delivered to the laboratory may be representative of the conditions to be evaluated, but it may not be in the proper physical form for the analytical procedure. Or, the sample may require drying, reduction in size, mixing, or some other form of treatment before analysis. The techniques utilized in this phase of sample preparation usually are routine.

Samples to be stored for some period of time before analysis often require special procedures. These procedures may include steps to prevent the loss of some radionuclides, or sample decomposition or spoilage. Procedures may vary from simple refrigeration to addition of acid to urine samples for the prevention of adsorption of trace elements on glass containers.

Polyethylene containers adsorb fewer elements in solution than does glass, and even these few can be prevented by the addition of acid, a carrier, or a complexing agent. Thus, the storage of most solutions presents a relatively minor problem.

Vegetation and other biological materials are not so simple. These organic materials are subject to spoilage; therefore, it is desirable to convert them to some more stable form as soon after collection as possible. Most of these materials can be stabilized by drying. Short-term storage may require only refrigeration. Liquids may be preserved by the addition of a few milliliters of formaldehyde.

SAMPLING OF VEGETATION

Vegetation samples may be used to determine the radiological history of an area or for determining areas of contamination. Only the leafy portions of samples usually are counted. The vegetation cover from at least one square meter of ground should be collected. The sample should contain at least one-half kilogram of material. The vegetation should be cut with shears at a height approximately one to two centimeters above the soil surface to simulate what a grazing animal would consume. The sample must be supported while it is cut; support prevents cross contamination by contact with the soil. The sample is placed into a 30- x 40-cm plastic bag. A bag of these dimensions approximately half full will contain approximately one-half kilogram of vegetation. Air is compressed from the bag, and the bag end is sealed.

The bag then is transported to the laboratory. If the sample is not to be examined immediately, it should be refrigerated. Samples that are to be kept for an extended period of time should be dried in accordance with standard plant-drying techniques.

SAMPLING OF LIQUID BIOLOGICAL MATERIAL

Liquid biological materials include such items as milk, urine, and blood. These materials should be collected directly into clean, contamination-free, well-stoppered glass or plastic containers when possible. When direct collection is impossible, the samples should be transferred to the sample bottles as quickly as possible, by use of clean techniques.

Blood samples should be treated with either heperin or sodium oxylate to prevent coagulation. Milk may have a few milliliters of formalin added as a preservative. All three materials should be packed in ice or refrigerated for transportation to the laboratory. Minimum sample sizes are as follows: milk, 1 liter; urine, 250 milliliters; and blood, 100 milliliters.

SAMPLING OF SOLID BIOLOGICAL MATERIAL

Solid biological materials other than vegetation include organ and tissue samples from animals, animal feces, bone, and hair. Samples of these materials should be collected in clean, contamination-free, tightly stoppered glass or plastic containers. They should be packed in ice or refrigerated immediately, then transported to the laboratory for analysis. Whole organs should be collected if possible; however, 10-grams is about the minimum useful quantity. Feces samples should be between 250 and 500 grams. (Note: to have a whole kilogram of any sample is better than not to have enough.)

FOOD SAMPLES

Foodstuffs should be collected in clean, contamination-free, tightly stoppered glass or plastic containers. Preservation is by the usual means of preserving the particular material; that is, prepared or spoilable material should be packed in ice or refrigerated, cereals and grains may be simply sealed in the container, etc. Minimum sample size should be 100 grams.

SAMPLE PREPARATION

After a sample reaches the laboratory it usually is measured in one of three ways prior to ashing, depending upon how the results are to be reported. It may be weighed immediately to determine net weight, dried and weighed to determine dry weight, or its volume may be measured to report activity per unit volume. In any case, the next step is the reduction of its volume by a process known as "ashing."

ASHING PROCEDURES

Three different procedures are used for reduction of the volume of organic material by ashing. These are dry ashing, wet ashing or acid digestion, and fusion.

Dry Ashing Explained

Dry ashing, although less time consuming than wet ashing, can be used only to prepare organic samples to be analyzed for nonvolatile radionuclides. Samples to be investigated for the presence of iodine, polonium, or other nuclides that are volatile at temperatures below 500°C must not be prepared in this manner.

Organ and tissue samples have a tendency to sell or splatter as they are dry-ashed, even when they have been dried "thoroughly." The technician can reduce sample loss due to splatter by using larger crucibles, by dissecting or homogenizing the sample before ashing, and by raising the furnace temperature in small steps until the final temperature is reached. The following are four typical dry ashing procedures.

Dry Ashing of Vegetation Samples

Leaf vegetation samples are dried at 105°C, then ashed in a muffle furnace at 550°C. The ash then is dissolved in hydrochloric acid for radiochemical analysis. The entire procedure is accomplished as follows:

Procedure:

1. Place 500 grams, wet weight, of the sample in a porcelain evaporating dish and dry at 105°C for 24 hours.
2. Transfer evaporating dish with the dry sample to a muffle furnace.
3. Bring temperature of the muffle furnace up to 550°C over a period of 1 hour.
4. Hold at 550°C for 5 hours.
5. Remove dish and sample from the muffle furnace and allow to air cool.
6. Add 75 ml of 6 N hydrochloric acid, and digest on a steam bath for 30 minutes.
7. Quantitatively transfer to a centrifuge tube, and centrifuge for 5 minutes.
8. Decant the supernate through a fiberlass filter, saving the filtrate. (Note: Leave the filtrate in the filter flask with the filter and filter assembly in place.)
9. Add 30 ml of concentrated hydrochloric acid to the residue in the centrifuge tube; stir.
10. Place in a boiling-water bath for 6 hours.

11. Filter through the same filter used previously, combining the filtrates.
12. Wash the residue 3 times with 10-ml portions of distilled water, filtering and combining the washings with the filtrates.
13. Quantitatively transfer to a volumetric flask, and dilute to a known volume.

Dry Ashing of Bone Samples

Bone samples are dry ashed for analysis of nonvolatile radionuclides. Since cesium-137 is volatile at the final ashing temperature, it should be determined by gamma counting of the 0.662-MeV photons after the first ashing procedure is completed. All extraneous tissue should be removed from the bone before the procedure is begun. Several samples may be processed at the same time since from two to three days usually are required to complete a preparation, with much waiting between operations. As outlined in the following procedure, the sample first is ashed at 250°C for 24 hours, ashed at 450°C for 3 hours, then ashed at 800°C for 24 hours.

Procedure:

1. Weigh a sample of bone in a crucible.
2. Place in a muffle furnace, and bring the temperature to 250°C for 24 hours.
3. Remove sample from the furnace, cool, and reweigh. If cesium-137 is to be determined, gamma count at this time.
4. Place the sample into a platinum crucible.

5. Place crucible and sample into a muffle furnace, and bring the temperature to 450°C for 3 hours.
6. After 3 hours, increase the temperature to 800°C, and hold for 24 hours.
7. Remove crucible and sample from the muffle furnace, allow to cool in desiccator, and reweigh.
8. Proceed with radiochemical analysis. (Note: The sample of ash can be stored if necessary.)

Dry Ashing of Blood Plasma Samples

Blood plasma or serum may be prepared for the analysis of nonvolatile radionuclides by this procedure. When the sample is whole blood, the cellular component should be allowed to settle and the plasma separated for analysis. The blood plasma first is dried at 105°C, then ashed at 300°C to destroy sugars. The residue then is ashed at 500°C to destroy remaining organic material, as outlined in the following procedure.

Procedure:

1. Place plasma in a pyrex dish; dry in an oven at 105°C for 48 hours.
2. Transfer sample to a large furnace, and slowly increase the temperature to 300°C over a period of 3 hours.
3. Maintain temperature at 300°C for 4 hours.
4. Quantitatively transfer the charred sample to a crucible.
5. Place the crucible and sample into a muffle furnace, and bring the temperature to 500°C.
6. Maintain temperature at 500°C for at least 48 hours.
7. Remove the sample, cool in a desiccator, and weigh.

8. The sample now is ready for radiochemical analysis. (Note: The sample can be stored as ash if necessary.)

Dry Ashing of Hair Samples

Hair samples may be ashed for nonvolatile radionuclide analysis in at least a three-step operation. Before the sample is ashed, it must be washed thoroughly at least 3 times with a good detergent and rinsed between each washing. The sample then is dried, weighed, and slowly ignited in a muffle furnace. The ignited sample finally is ashed at 600°C, as in the below procedure.

Procedure:

1. Dry a clean sample of hair in a pyrex container in a drying oven at 105°C for 48 hours.
2. Weigh the clean, dry sample; record the weight as the original weight of the sample. (Note: Samples up to 1000 grams may be used.)
3. Place the weighed sample into a pyrex container, and place in an electric oven.
4. Slowly raise the temperature to 300°C, stopping and holding the temperature constant when any smoking occurs, until the smoking stops. (Note: The first smoking should occur at approximately 270°C.)
5. Maintain the temperature at 300°C for 1 hour.
6. Remove from the oven, cool, and quantitatively transfer to a crucible.
7. Place the crucible and sample into a muffle furnace, and ash at 600°C for 48 hours.
8. Remove the crucible and sample from the furnace, cool in a desiccator, and weigh. (Note: The completely

ignited sample will be white to light tan in color and will be extremely light and fluffy. Care must be taken to prevent air currents from removing some of the sample.)

9. The sample now is ready for radiochemical analysis.

Wet Ashing Explained

Samples that contain radionuclides that are volatile at dry-ashing temperatures are placed in solution by acid digestion or wet ashing techniques. Variations on the Kjeldahl procedure that employs concentrated sulfuric acid have been widely used. Combinations of nitric, sulfuric, hydrochloric, and perchloric acids along with hydrogen peroxide are satisfactory. WARNING: Perchloric acid mixtures are extremely explosive with organic materials. Solutions that contain perchloric acid must never be allowed to dry. The advantage of wet ashing over dry ashing is that there are fewer chances for loss due to volatilization, fusion with crucibles, adsorption onto silica residues, loss of ash, etc.

Wet Ashing of Vegetation Samples

Samples of vegetation to be examined for volatile radionuclides are placed in solution by digestion in nitric acid. Silica is solubilized with hydrofluoric acid, and the remaining organic matter is dissolved with perchloric acid. NOTICE: Perchloric acid treatment must be performed under a perchloric acid hood.

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Procedure:

1. Weight 500 grams of vegetation, wet weight. (Note: Dry samples may be used, but be sure to note whether the sample weight is wet or dry.)
2. Place the sample in a large beaker.
3. Add 600 ml of concentrated nitric acid. (Note: If the quantity of acid is not sufficient to cover the sample, add additional acid until the sample is covered.)
4. Place the beaker with the sample onto a hot plate under a fume hood; place a mechanical stirrer in the solution.
5. Boil gently, with continuous slow stirring, until the acid solution evaporates to a volume of approximately 100 ml.
6. Add 400 ml of fuming nitric acid, and continue to boil with continuous stirring until the solution evaporates to a volume of approximately 150 ml.
7. Quantitatively transfer the solution and precipitate to a 250-ml centrifuge flask, using distilled water.
8. Centrifuge for 15 minutes.
9. Decant into a 600-ml beaker, saving the decantate.
10. Quantitatively transfer the precipitate to a platinum evaporating dish.
11. Add 25 ml of concentrated hydrofluoric acid to the precipitate in the platinum dish, and slowly evaporate to dryness on a hot plate under the fume hood.
12. Cool, then add 30 ml of hydrofluoric acid and again evaporate to dryness.
13. Repeat Step 12 until no more silica remains.
14. Add 10 ml of distilled water and 10 ml of concentrated nitric acid, and gently boil for 10 minutes.
15. Cool and quantitatively transfer to the decantate from Step 9.

WARNING: THE FOLLOWING STEPS MUST BE PERFORMED UNDER A PERCHLORIC ACID FUME HOOD! NO EXCEPTIONS! READ NOTICE ABOUT SAFETY EQUIPMENT IN STEP 16.

16. Add 20 ml of concentrated perchloric acid to the solution in the beaker, and carefully evaporate the solution on a hot plate until large volumes of perchloric acid fumes are evolved. (WARNING: DO NOT EVAPORATE TO DRYNESS. The solution should be a light brown color. Violent frothing or explosion may occur while evaporating nitric/perchloric acid/organic mixtures. Wear safety equipment and work behind a safety shield at all times. If frothing occurs, or if the solution suddenly turns dark, remove the sample from the hot plate, and evacuate the area quickly. After the sample has cooled, return, add 10 ml of concentrated nitric acid, and continue fuming.)
17. Continue fuming until color of the solution changes to a light yellow-green.
18. When the solution is cool, it is ready for radiochemical analysis.

Wet Ashing of Soft Tissue Samples

Samples from organs or tissues are prepared by this wet ashing technique for radiochemical analysis of elements that are volatile above 200°C. Samples to be treated by this technique are best preserved by freezing. After the sample has been defrosted, it is dissected and dried at 105°C. The organic material is solubilized by nitric acid digestion, and silica is removed by hydrofluoric acid treatment. The residue is dissolved in hydrofluoric acid before analysis.

Procedure:

1. If the sample has been preserved by freezing, defrost it by allowing it to stand at room temperature overnight.
2. Dissect as much fat as possible from organs. Cut the sample into approximately 5-mm cubes, puncturing any membranes that could retain gases or vapors during heating. Remove any lymph nodes present.
3. Place the sample in a pre-weighed evaporating dish, and dry in a drying oven at 105°C for 24 hours.
4. Determine the dry weight of the sample.
5. Place the sample in a 1000-ml beaker, and slowly add 250 ml of concentrated nitric acid.
6. Place the beaker on a hot plate, and bring the liquid to a slow boil. Continue heating until the sample is dissolved.
7. Evaporate the solution to a volume of approximately 50 ml.
8. Add 100 ml of fuming nitric acid to the sample, and again evaporate to a volume of approximately 50 ml.
9. Repeat Step 8 until the solution is a light brown color.
10. Quantitatively transfer the solution to a 250-ml Teflon beaker.
11. Add 20 ml of concentrated hydrofluoric acid, and evaporate to a volume of approximately 30 ml.
12. Wash down the sides of the beaker with 10 ml of concentrated hydrchloric acid, and evaporate to a volume of approximately 30 ml.
13. Repeat Step 12 three more times.
14. Add 5 ml of concentrated hydrochloric acid and 15 ml of distilled water.

15. Quantitatively transfer the solution to a 100-ml volumetric flask, and dilute to the mark with distilled water.
16. The solution is now ready for radiochemical analysis.

Liquid Biological Samples Explained

Liquid biological samples may be urine, blood, plasma, gastric liquids, or any of a number of other fluids found in living organisms. Regardless of the origin of the liquid, this procedure may be utilized to prepare the sample for analysis. This method has been used with considerable success for bioassay procedures.

The sample is evaporated to dryness and the organic matter digested with nitric acid and hydrogen peroxide. The salts then are dissolved in hydrochloric acid for analysis. (Note: This procedure may not be used for volatile radionuclides.)

Procedure:

1. Place 1000 ml of the sample in a 2-liter beaker, and slowly evaporate to dryness under a fume hood. Use a hot plate as a source of heat.
2. Carefully add 500 ml of concentrated nitric acid, and again evaporate to dryness. (Note: When the volume of solution reaches approximately 100 ml, reduce the rate of evaporation to a very slow rate.)
3. Add 100 ml of concentrated nitric acid and 5 ml of 30% hydrogen peroxide, and evaporate to dryness.
4. Repeat Step 3 two times.
5. Add 30 ml of concentrated nitric acid and 2 ml of 30% hydrogen peroxide. Place on a thermostatically con-

trolled hot plate, and evaporate to dryness. Bake for 2 hours at 180°C.

6. Add 100 ml of distilled water to dissolve the residue.
7. Repeat Step 6 three times.
8. Dissolve the residue in 100 ml of 0.5 N hydrochloric acid. Heat gently, if necessary, to dissolve all the solids.
9. Allow to cool to room temperature.
10. The sample is now ready for radiochemical analysis.

FUSION PREPARATIONS

Samples not readily dissolved by acid digestion may be put into solution by the fusion technique. Samples with high silica content nearly always require fusion. This is particularly true of soil and silt samples, but may be applied to ashed food, vegetation, or biological materials. Most ashed samples that are not readily soluble in acid will go into solution quite easily after fusion.

This procedures does not require great experience or expertise, and most technicians should be able to process several samples per day.

Carrier solutions for the radiochemical analyses to be performed are added to the sample, and the mixture is fused with sodium hydroxide and sodium carbonate. After fusion, the residue is dissolved in dilute hydrochloric acid for radiochemical analysis.

Procedure:

1. Place 5 grams of the sample in a 250-ml nickel crucible.
2. Add appropriate quantities of carrier solutions for the particular radiochemical analyses to be performed.

3. Add 5 grams of sodium hydroxide pellets for each gram of sample in the mixture, and mix thoroughly with a nickel spatula.
4. Fuse over a Meeker burner for 30 minutes.
5. Slowly stir in 0.5 g of anhydrous sodium carbonate for each gram of the original sample.
6. Continue heating for 30 minutes.
7. Remove the flame, and allow the fused mixture to cool.
8. Add 200 ml of hot distilled water, and bring to a gentle boil to break up the solid.
9. Quantitatively transfer to a 250-ml centrifuge flask.
10. Centrifuge for 5 minutes, then decant, discarding the decantate.
11. Add 40 ml of 3 N sodium carbonate, and place in a boiling-water bath for 10 minutes.
12. Centrifuge for 5 minutes, and decant, discarding the decantate..
13. Dissolve the precipitate in a minimum quantity of 6 N hydrochloric acid.
14. Quantitatively transfer the solution to a 250-ml volumetric flask, using distilled water, and dilute to volume.
15. The sample is now prepared for radiochemical analysis.

PREPARATION OF CARRIER SOLUTIONS

BARIUM

A solution that contains 1 mg of Ba^{+2} per milliliter may be prepared: 1.9 grams of barium nitrate is dissolved in 900 ml of distilled water; 1 ml of 16 N nitric acid is added; and the solution is diluted to 1 liter. More concentrated solutions may be prepared if the amount of barium nitrate used is increased – the quantity being based upon multiples of 1.9 grams times the number of milligrams per milliliter desired.

BISMUTH

The technician can prepare a solution containing 1 mg of Bi^{+3} per ml by dissolving 1.113 grams of bismuth oxide in 2 N hydrochloric acid and diluting to 1 liter with 2 N hydrochloric acid. More concentrated solutions can be prepared in the same manner as the barium solutions.

CALCIUM

A solution that contains 1 mg of Ca^{+2} per ml can be prepared by the dissolving of 5.8925 grams of calcium nitrate, $Ca(NO_3)_2 \cdot 4\ H_2O$ in 900 ml of distilled water, by the addition of 1 ml of 16 N nitric acid and dilution to 1 liter with distilled water. More concentrated solutions can be prepared in the same manner as the barium solutions.

IRON

A solution that contains 1 mg of Fe^{+3} per ml can be prepared as follows: Dissolve 4.84 grams of ferric chloride, $FeCl_3 \cdot 6\ H_2O$, in 900 ml of distilled water; add 1 ml of 16 N nitric acid; dilute to 1 lister with distilled water. More concentrated solutions can be prepared in the same manner as the barium solutions.

LANTHANUM

A solution that contains 10 mg of La^{+3} per ml can be prepared in the following manner:

1. Dissolve 3.1 gm of lanthanum nitrate, $La(NO_3)\cdot 6\ H_2O$, in 50 ml of distilled water in a 100-ml centrifuge tube.
2. Add 30 ml of concentrated ammonium hydroxide, stir, and centrifuge for 10 minutes. Decant, discarding the decantate.
3. Add 75 ml of 6 N ammonium hydroxide, stir, and centrifuge for 5 minutes. Decant, discarding the decantate.
4. Repeat Step 3.
5. Add 75 ml of distilled water, stir, and centrifuge for 5 minutes. Decant, discarding the decantate.
6. Dissolve the precipitate in a minimum amount of concentrated nitric acid, and dilute to 50 ml with distilled water.
7. Repeat Steps 2 through 5.
8. Quantitatively transfer the precipitate to a 250-ml beaker, using distilled water.
9. Evaporate the liquid to dryness.
10. Cool and dissolve the solid in 100 ml of distilled water.
11. Transfer the solution to a 125-ml separatory funnel.
12. Add 20 ml of a solution of 100 g of 2-Thenoyltrifluoroacetone per liter of benzene, shake for 10 minutes, and allow the phases to separate.

13. Drain the aqueous layer through a Whatman No.542 filter paper, into a glass-stoppered reagent bottle.
14. Check the purity of the solution by evaporating 1 ml of the solution in a stainless steel planchet and by counting the alpha activity. An activity of less than 0.3 dpm is an acceptable purity.
15. The solution will contain approximatley 10 mg La^{+3} per milliliter. Solutions of greater concentration can be prepared if the quantity of lanthanum in Step 1 is increased.

LEAD

To prepare a solution that contains 1 mg of Pb^{+2} per ml, dissolve 1.599 g of lead nitrate in distilled water and dilute to 1 liter. More concentrated solutions are prepared in the same manner as the barium solutions.

STRONTIUM

A solution that contains 1 mg of Sr^{+2} per ml can be prepared as follows: Dissolve 2.415 grams of strontium nitrate in 900 ml of distilled water; add 1 ml of 16 N nitric acid; and dilute to 1 liter with distilled water. More concentrated solutions are prepared in the same manner as the barium solutions.

YTTRIUM

The technician can prepare a solution that contains 10 mg of Y^{+3} per ml by dissolving 12.7 grams of yttrium oxide in 50 ml of warm, 16 N nitric acid, diluting to 900 ml with distilled water, adjusting to a pH of 2 with concentrated ammonium hydroxide, and diluting to 1000 ml with distilled water.

EXERCISES

1. Outline a sampling procedure for vegetation sampling.
2. What is the difference between wet and dry ashing?
3. Should a sample to be analyzed for a volatile radionuclide be solubilized by wet or dry ashing? List the reasons.
4. Why should perchloric acid treatment be performed only under a perchloric acid fume hood?
5. Why should a sample of humus be treated by the fusion method rather than dry ashing?
6. Outline the procedure for the preparation of a sample of material from the liver of an animal for analysis of a nonvolatile radionuclide.
7. Outline the procedure for the preparation of a sample of urine for the analysis of a volatile radionuclide.
8. Write the procedure for the preparation of a milk sample for the analysis of ^{90}Sr.
9. List the general criteria for sample collection.
10. Why should a portion of all samples be stored for future use?

11. Why are polyethylene containers preferred to glass containers?
12. Outline the procedure for the wet ashing of a vegetation sample.

MATERIALS

500 g cigarette tobacco.
Evaporating dish.
Drying oven.
Muffle furnace.
Hot plate.
Glass fiber filters.
Suction filter apparatus.
Centrifuge.
100-ml centrifuge tubes.
Water bath.
Hydrochloric acid.
Ascorbic acid.
Stirring motor.
Glass stirring rod.
Heat lamp.
Silver planchets.
Plating cell for polonium-210.
Alpha counter.
100 ml of milk spiked with 0.005 µCi of ^{90}Sr.
Strontium carrier solution.
1-liter beaker.
Concentrated nitric acid.
Magnetic stirring hot plate.

Fuming nitric acid.
250-ml separatory funnel.

PROCEDURES

RADIOASSAY OF TOBACCO FOR ^{210}Po

1. Collect approximately 500 grams of cigarette tobacco.
2. Weight the sample.
3. Place in a preweighed evaporating dish, and dry in a drying oven for 24 hours at 105°C.
4. Cool in a desiccator to room temperature.
5. Weigh the sample.
6. Place the evaporating dish with the sample into a muffle furnace, and bring the temperature up to 550°C over a period of 4 hours.
7. Maintain at 550°C for 5 hours.
8. Remove the dish that now contains ash from the furnace, and allow to cool.
9. Add 75 ml of 6 N hydrochloric acid, and heat on a hot plate for 5 minutes.
10. Quantitatively transfer the material to a 100-ml centrifuge tube, and centrifuge for 5 minutes.
11. Decant the liquid in the centrifuge tube through a glass fiber filter, using suction. Save both the decantate and the precipitate.
12. Slurry the precipitate in the centrifuge tube with 30 ml of concentrated hydrochloric acid, and place in a boiling-water bath for 4 hours.

13. Filter the solution through the same filter used previously, combining the filtrates.
14. Wash the residue in the centrifuge tube three times with distilled water, filtering through the same filter used previously, combining the filtrates.
15. Quantitatively transfer the filtrates to an evaporating dish and evaporate to dryness. NOTICE: DO NOT BAKE!
16. Dissolve the residue in 150 ml of 0.5 N hydrochloric acid.
17. Add 1 gram of ascorbic acid.
18. Quantitatively transfer the solution to a polonium plating cell equipped with a silver disc (illustrated in Figure 20-7-1).
19. Place the plating cell in a 95°C water bath.

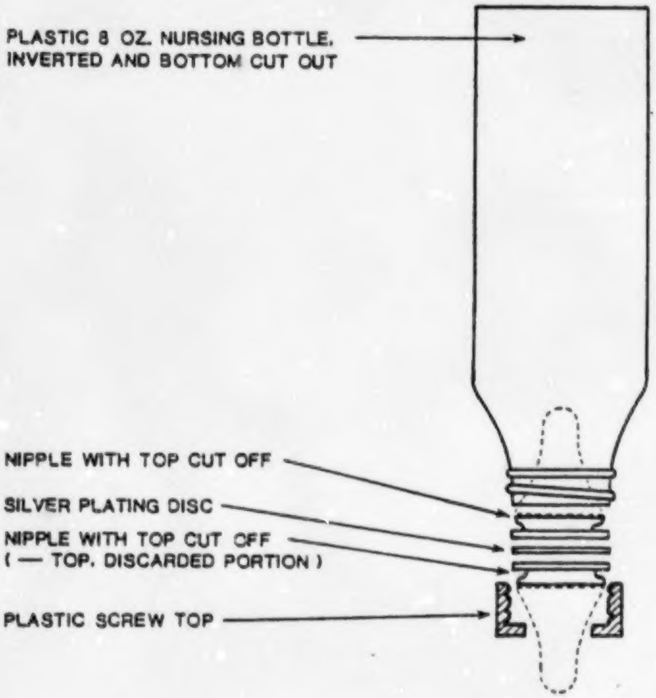
PLASTIC 8 OZ. NURSING BOTTLE, INVERTED AND BOTTOM CUT OUT
NIPPLE WITH TOP CUT OFF
SILVER PLATING DISC
NIPPLE WITH TOP CUT OFF (— TOP, DISCARDED PORTION)
PLASTIC SCREW TOP

Figure 20-7-1. A plating cell for polonium-210 determination.

20. Stir the solution with a motor-drive glass stirring rod.

21. Quantitatively transfer the solution to a 400-ml beaker, using 0.5 N hydrochloric acid.

22. Remove the silver disc, wash with distilled water, and dry under a heat lamp.

23. Cool to room temperature, and count the alpha activity with an alpha counter.

24. Calculate the activity of the ^{210}Po according to the following equation:

$$\text{Activity } ^{210}\text{Po (pCi)} = \frac{C}{E \times P} \qquad \text{Equation 20-7-1 (Equation = Eq.)}$$

where:

C = alpha count rate.

E = counting efficiency for ^{210}Po plated on a silver disc

P = plating efficiency, experimentally found to be 95% for this technique.

25. Record data in Data Table 20-7-1.

ANALYSIS OF ^{89}Sr and ^{90}Sr IN MILK

1. Place 100 ml of a sample of milk in a 250-ml separatory funnel.
2. Add 2 ml of a carrier solution containing 10 mg of Sr^{+2} per ml, stopper, and shake for 1 minute.
3. Place 300 ml of concentrated nitric acid in a 1-liter beaker; place on a magnetic stirring hot plate; and heat to boiling.

4. Place the separatory funnel containing the milk sample on a ring stand so that it is suspended above the beaker of boiling nitric acid.
5. Adjust the stopcock on the separatory funnel in such a manner that the milk is added to the constantly-stirred, boiling nitric acid at a rate of approximately 2 drops per second.
6. Rinse the separatory funnel 3 times with distilled water, adding the rinsings to the boiling acid.
7. Remove the stirring bar, and evaporate the solution to dryness.
8. Add 100 ml of fuming nitric acid, and evaporate to dryness.
9. Add 50 ml of nitric acid, and evaporate to dryness.
10. Take up the residue with approximately 25 ml of 5 N nitric acid; and filter, with suction, through a glass fiber filter, saving the filtrate.
11. Analyze the sample according to the procedure given for strontium-89 and -90 in Module 20-5, beginning with Step 4.
12. Record data in Data Table 20-7-2.

DATA TABLES

DATA TABLE 20-7-1
RADIOASSAY OF TOBACCO FOR POLONIUM-210

Sample	Wet Weight	Dry Weight	Net Alpha Count Rate	Activity as Polonium-210

DATA TABLE 20-7-2
ANALYSIS OF ^{89}Sr and ^{90}Sr IN MILK

Sample	Net Beta Count Rate	Activity per 100 ml	Activity per liter
Y for ^{90}Sr			
Total			
^{89}Sr			

REFERENCE MATERIALS

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IAEA, Environmental Monitoring in Emergency Situations. New York: Unipub, Inc., 1966.

TEST

1. Write the procedures to be observed in sampling of vegetation.

2. Outline a procedure for dry ashing a vegetation sample.

3. Outline a procedure for wet ashing a vegetation sample.

4. Outline a procedure for the analysis of a sample of milk for the presence of ^{90}Sr.

5. Why are samples to be analyzed for volatile radionuclides usually not prepared by dry ashing?

NUCLEAR TECHNOLOGY

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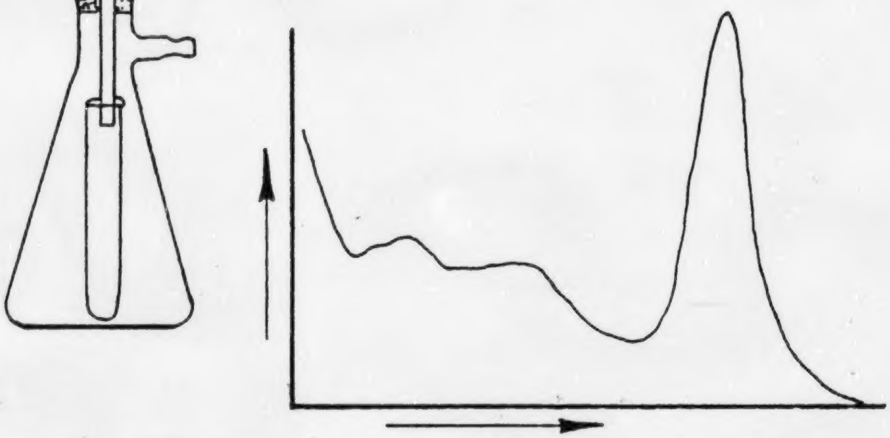
RADIATION MONITORING
TECHNIQUES (RADIOCHEMICAL)

COURSE 20



CORD CENTER FOR OCCUPATIONAL RESEARCH AND DEVELOPMENT

NUCLEAR TECHNOLOGY SERIES
INDEX
for
COURSE 20, RADIATION MONITORING TECHNIQUES (RADIOCHEMICAL) (SEVEN MODULES)

NOTE: Each module in this Course is numbered separately, and the page numbers begin with page 1 in each module. Therefore, this Index lists for each entry the module number (underlined), followed by a comma and the page number. Figures and tables are listed by their numbers, followed by the module and page number in parentheses. A Master Index is available in a separate volume. This general index merges all index entries from the 35 Nuclear Courses. It is a valuable research tool, with which the student or researcher can locate all the references to any subject within the 35 Courses.

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